

(18)



Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) Publication number:

**0 070 698
B1**

(12)

EUROPEAN PATENT SPECIFICATION

(45) Date of publication of patent specification: 13.11.85

(51) Int. Cl.⁴: **C 07 D 403/12**, A 01 N 47/36
// C07D209/30

(21) Application number: **82303730.4**

(22) Date of filing: **15.07.82**

(54) **Herbicidal indole sulfonamides.**

(30) Priority: 16.07.81 US 283928
01.06.82 US 382876

(43) Date of publication of application:
26.01.83 Bulletin 83/04

(45) Publication of the grant of the patent:
13.11.85 Bulletin 85/46

(84) Designated Contracting States:
AT BE CH DE FR GB IT LI LU NL SE

(58) References cited:
EP-A-0 039 239
EP-A-0 045 196
US-A-4 127 405

(73) Proprietor: **E.I. DU PONT DE NEMOURS AND
COMPANY**
1007 Market Street
Wilmington Delaware 19898 (US)

(72) Inventor: **Zimmerman, Donna Frieze**
R.D.1 Box 200 A
Landenberg Pennsylvania 19350 (US)

(74) Representative: **Hildyard, Edward Martin et al**
Frank B. Dehn & Co. European Patent Attorneys
Imperial House 15-19 Kingsway
London WC2B 6UZ (GB)

The file contains technical information
submitted after the application was filed and
not included in this specification

Note: Within nine months from the publication of the mention of the grant of the European patent, any person may give notice to the European Patent Office of opposition to the European patent granted. Notice of opposition shall be filed in a written reasoned statement. It shall not be deemed to have been filed until the opposition fee has been paid. (Art. 99(1) European patent convention).

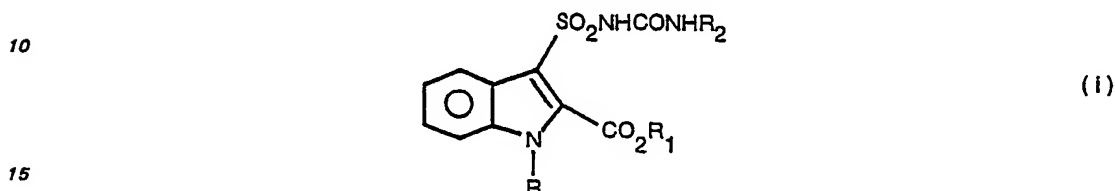
EP 0 070 698 B1

Description

Background of the Invention

This invention relates to novel N-(heterocyclicaminocarbonyl)indole sulfonamides which are useful as agricultural chemicals and, in particular, as herbicidal agents.

Canadian Patent 747,920 (issued 12/6/66, Upjohn) discloses 3-(alkylcarbamoysulfamoyl)-1-alkylindole-2-carboxylic acids and esters (i) as sedatives, diuretics, antifungal agents and/or sun screens

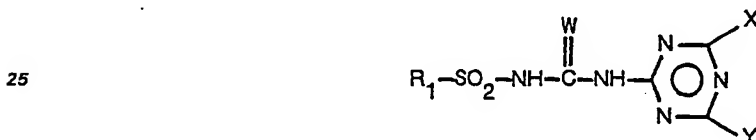


where

R and R₂ are C₁—C₄ alkyl, and

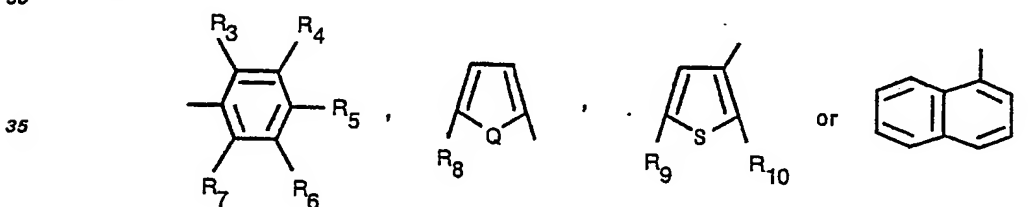
R₁ is H or C₁—C₄ alkyl.

20 U.S Patent 4,127,405 teaches compounds which are useful for controlling weeds in wheat having the formula



wherein

30 R₁ is



40 R₃ and R₆ are independently hydrogen, fluorine, chlorine, bromine, iodine, alkyl of 1—4 carbon atoms, alkoxy of 1—4 carbon atoms, nitro, trifluoromethyl, cyano, CH₃S(O)_n— or CH₃CH₂S(O)_n—;

R₄ is hydrogen, fluorine, chlorine, bromine or methyl;

R₅ is hydrogen, fluorine, chlorine, bromine, methyl or methoxy;

45 R₇ is hydrogen, fluorine, chlorine, bromine, alkyl of 1—2 carbon atoms or alkoxy of 1—2 carbon atoms;

R₈ is hydrogen, methyl, chlorine or bromine;

R₉ and R₁₀ are independently hydrogen, methyl, chlorine or bromine;

W and Q are independently oxygen or sulfur;

n is 0, 1 or 2;

50 X is hydrogen, chlorine, bromine, methyl, ethyl, alkoxy of 1—3 carbon atoms, trifluoromethyl, CH₃S— or CH₃OCH₂—; and

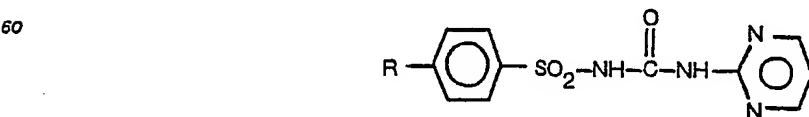
Y is methyl or methoxy; or their agriculturally suitable salts; provided that:

(a) when R₅ is other than hydrogen, at least one of R₃, R₄, R₆ and R₇ is other than hydrogen and at least two of R₃, R₄, R₆ and R₇ must be hydrogen;

55 (b) when R₅ is hydrogen and all of R₃, R₄, R₆ and R₇ are other than hydrogen, then all of R₃, R₄, R₆ and R₇ must be either chlorine or methyl; and

(c) when R₃ and R₇ are both hydrogen, at least one of R₄, R₅ or R₆ must be hydrogen.

French Patent No. 1,468,747 discloses the following *para*-substituted phenylsulfonamides, useful as antidiabetic agents:



65 wherein R = H, halogen, CF₃ or alkyl.

0 070 698

Logemann et al. Chem. Ab., 53, 18052 g (1959), disclose a number of sulfonamides, including uracil derivatives and those having the formula:

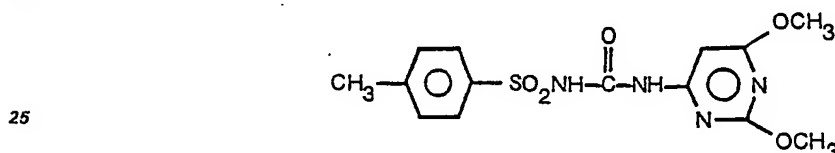


wherein R is butyl, phenyl or



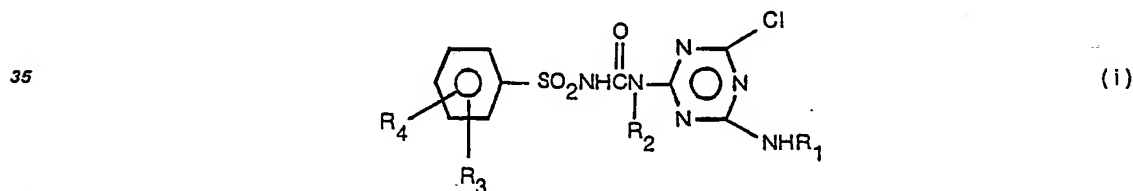
and R₁ is hydrogen or methyl. When tested for hypoglycemic effect in rats (oral doses of 25 mg/100 g), the compounds in which R is butyl and phenyl were most potent. The others were of low potency or inactive.

Wojciechowski, J. Acta. Polon. Pharm. 19, p. 121—5 (1962) [Chem Ab., 59 1633 e] describes the synthesis of N-[(2,6-dimethoxypyrimidin-4-yl)aminocarbonyl]-4-methylbenzenesulfonamide:



Based upon similarity to a known compound, the author predicted hypoglycemic activity for the foregoing compound.

30 Netherlands Patent 121,788, published September 15, 1966, teaches the preparation of compounds of Formula (i), and their use as general or selective herbicides,

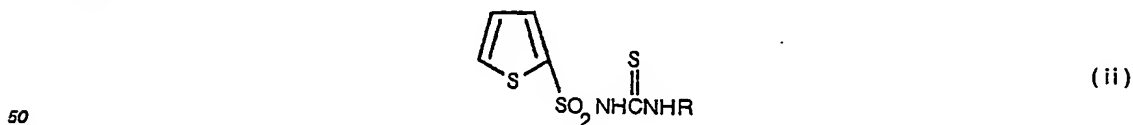


wherein

R₁ and R₂ may independently be alkyl of 1—4 carbon atoms; and

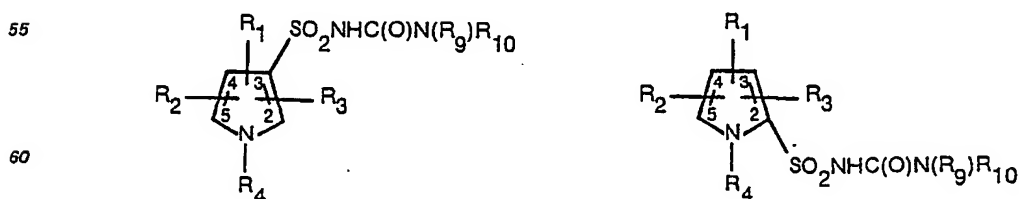
R₃ and R₄ may independently be hydrogen, chlorine or alkyl of 1—4 carbon atoms.

Compounds of Formula (ii), and their use as antidiabetic agents, are reported in *J. Drug. Res.* 6, 123 (1974),



wherein R is pyridyl.

U.S. Patent application 244,172 discloses herbicidal compounds of formulae

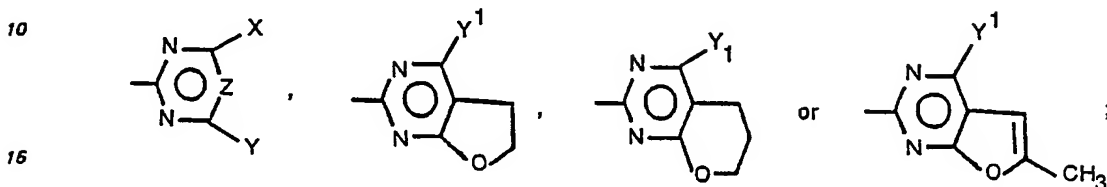


wherein

R₁ is H, C₁—C₄ alkyl, NO₂, CN, C(O)CCl₃, SO₂R₁₁, C(O)R₅ or CO₂H;

65 R₂ is H or C₁—C₄ alkyl;

R_3 is H, C_1-C_4 alkyl, Cl or Br;
 R_4 is H, C_1-C_4 alkyl, cyanoethyl, C_5-C_6 cycloalkyl, benzyl, phenyl substituted with Cl or NO_2 , or $C(O)R_6$;
 R_5 is C_1-C_4 alkyl or C_1-C_4 alkoxy;
 R_6 is C_1-C_4 alkyl, C_1-C_4 alkoxy, or NR_7R_8 ;
 R_7 and R_8 are independently C_1-C_2 alkyl;
 R_9 is H, CH_3 or OCH_3 ;
 R_{11} is C_1-C_4 alkyl;
 R_{10} is



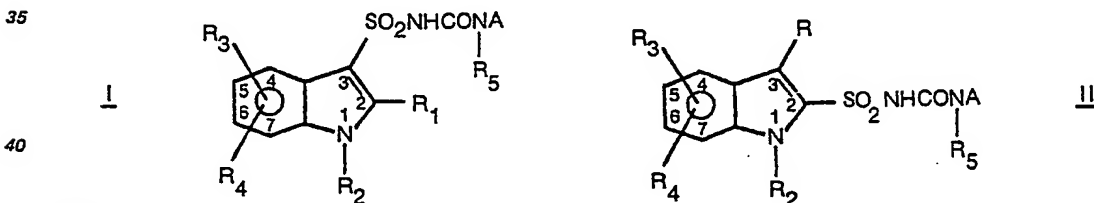
X is CH_3 or OCH_3 ;
Y is H, CH_3 , OCH_3 , OCH_2CH_3 , OCH_2CF_3 , Cl, CH_2OCH_3 , $CH_2OCH_2CH_3$ or CF_3 ;
 Y^1 is H, CH_3 , OCH_3 , Cl or OCH_2CH_3 ; and

20 Z is CH, N, CCH_3 , CCH_2CH_3 , CCH_2CH_2Cl , CCl, CBr or CF; and their agriculturally suitable salts.

The presence of undesired vegetation causes substantial damage to useful crops, especially agricultural products that satisfy man's basic food and fiber needs, such as cotton, rice, corn, wheat, and the like. The current population explosion and concomitant world food and fiber shortage demand improvements in the efficiency of producing these crops. Prevention or minimizing the loss of a portion of such valuable crops by killing, or inhibiting the growth of undesired vegetation is one way of improving this efficiency. A wide variety of materials useful for killing, or inhibiting (controlling) the growth of undesired vegetation is available; such materials are commonly referred to as herbicides. The need still exists however, for more effective herbicides.

30 Summary of the Invention

This invention relates to novel compounds of Formula I and Formula II, suitable agricultural compositions containing them, and their method of use as general, as well as selective, pre-emergence or post-emergence herbicides.



where

45 R is H, C_1-C_4 alkyl, $(CH_2)_mCO_2R_9$, $CH_2OC_2H_5$, SO_2R_{10} , CHO, $SO_2NR_{11}R_{12}$, $CH_2N(CH_3)_2$ or CH_2OCH_3 ;
 R_1 is H, C_1-C_4 alkyl, CO_2R_6 , $C(O)NR_7R_8$, $C(O)R_{10}$, SO_2R_{10} , or $SO_2NR_{11}R_{12}$;

R_2 is H, C_1-C_3 alkyl or $SO_2C_6H_5$;

R_3 is H, F, Cl, Br, C_1-C_3 alkyl, C_1-C_3 alkoxy or NO_2 ;

R_4 is H, Cl or Br;

R_5 is H or CH_3 ;

50 R_6 is C_1-C_4 alkyl, C_3-C_4 alkenyl, CH_2CH_2Cl or $CH_2CH_2OCH_3$;

R_7 and R_8 are independently H or C_1-C_4 alkyl, provided that the total number of carbon atoms is less than or equal to 4;

R_9 is H or C_1-C_3 alkyl;

R_{10} is C_1-C_3 alkyl;

55 R_{11} and R_{12} are independently C_1-C_3 alkyl, provided that the total number of carbon atoms is less than or equal to 4.

m is 0, 1 or 2;

A is



X is CH_3 or OCH_3 ;

Y is CH₃, OCH₃, OC₂H₅, CH₂OCH₃, Cl, H, C₂H₅ or N(CH₃)₂; and
Z is CH or N;

provided that

- 1) when R₂ is SO₂C₆H₅, then R₁ and R are H or C₁—C₄ alkyl.
- 2) when Y is Cl, then Z is CH; and
- 3) when m is 0, then R₉ is C₁—C₃ alkyl.

Preferred Compounds

Preferred for reasons of higher herbicidal activity and/or more favorable ease of synthesis are:

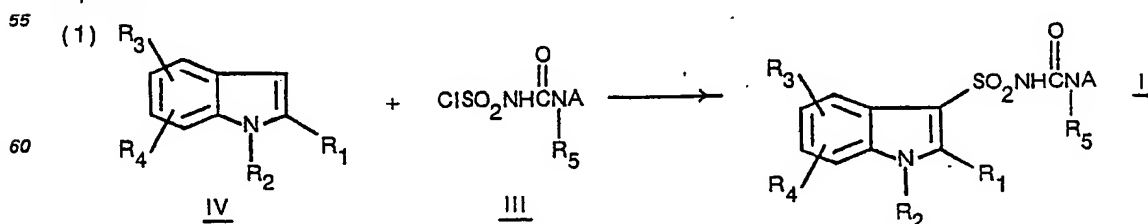
- (1) Compounds of the generic scope of Formula I where R₁ is H, C₁—C₃ alkyl, CO₂R₆, C(O)NR₇R₈, SO₂NR₁₁R₁₂ or SO₂R₁₀; and R₅ is H;
- (2) Compounds of Preferred (1) where R₃ and R₄ are H;
- (3) Compounds of Preferred (2) where R₂ is H or CH₃;
- (4) Compounds of Preferred (3) where R₁ is H, CO₂CH₃, SO₂CH₃ or SO₂N(CH₃)₂;
- (5) Compounds of Preferred (4) where Y is CH₃, OCH₃, OC₂H₅, CH₂OCH₃ or Cl;
- (6) Compounds of the generic scope of Formula II where R is H, C₁—C₃ alkyl, (CH₂)_mCO₂R₉, SO₂R₁₀ or SO₂NR₁₁R₁₂; and R₅ is H;
- (7) Compounds of Preferred (6) where R₃ and R₄ are H;
- (8) Compounds of Preferred (7) where R₂ is H or CH₃;
- (9) Compounds of Preferred (8) where R is H, CH₃ or (CH₂)_mCO₂—(C₁—C₃ alkyl); and
- (10) Compounds of Preferred (9) where Y is CH₃, OCH₃, OC₂H₅, CH₂OCH₃ or Cl.

Specifically Preferred for highest herbicidal activity and/or most favorable ease of synthesis are:

- 3-[[[4,6-dimethoxypyrimidin-2-yl]aminocarbonyl]aminosulfonyl]-1-methyl-1H-indole-2-carboxylic acid, methyl ester;
- 3-[[[4,6-dimethylpyrimidin-2-yl]aminocarbonyl]aminosulfonyl]-1-methyl-1H-indole-2-carboxylic acid, methyl ester;
- 3-[[[4-methoxy-6-methylpyrimidin-2-yl]aminocarbonyl]aminosulfonyl]-1-methyl-1H-indole-2-carboxylic acid, methyl ester;
- 3-[[[4,6-dimethoxy-1,3,5-triazin-2-yl]aminocarbonyl]aminosulfonyl]-1-methyl-1H-indole-2-carboxylic acid, methyl ester;
- 3-[[[4,6-dimethyl-1,3,5-triazin-2-yl]aminocarbonyl]aminosulfonyl]-1-methyl-1H-indole-2-carboxylic acid, methyl ester;
- 3-[[[4-methyl-6-methoxy-1,3,5-triazin-2-yl]aminocarbonyl]aminosulfonyl]-1-methyl-1H-indole-2-carboxylic acid, methyl ester;
- N-[[[4,6-dimethoxypyrimidin-2-yl]aminocarbonyl]-3-methyl-1H-indole-2-sulfonamide;
- N-[[[4,6-dimethylpyrimidin-2-yl]aminocarbonyl]-3-methyl-1H-indole-2-sulfonamide;
- N-[[[4-methyl-6-methoxypyrimidin-2-yl]aminocarbonyl]-3-methyl-1H-indole-2-sulfonamide;
- N-[[[4,6-dimethoxy-1,3,5-triazin-2-yl]aminocarbonyl]-3-methyl-1H-indole-2-sulfonamide;
- N-[[[4,6-dimethyl-1,3,5-triazin-2-yl]aminocarbonyl]-3-methyl-1H-indole-2-sulfonamide;
- N-[[[4-methyl-6-methoxy-1,3,5-triazin-2-yl]aminocarbonyl]-3-methyl-1H-indole-2-sulfonamide;
- 3-[[[4,6-dimethoxypyrimidin-2-yl]aminocarbonyl]aminosulfonyl]-1H-indole-2-carboxylic acid, ethyl ester;
- 3-[[[4,6-dimethoxy-1,3,5-triazin-2-yl]aminocarbonyl]aminosulfonyl]-1H-indole-2-carboxylic acid, ethyl ester;
- 3-[[[4-methoxy-6-methyl-1,3,5-triazin-2-yl]aminocarbonyl]aminosulfonyl]-1H-indole-2-carboxylic acid, ethyl ester;
- 3-[[[4-methoxy-6-methylpyrimidin-2-yl]aminocarbonyl]aminosulfonyl]-1H-indole-2-carboxylic acid, ethyl ester;
- 3-[[[4,6-dimethylpyrimidin-2-yl]aminocarbonyl]aminosulfonyl]-1H-indole-2-carboxylic acid, ethyl ester.

Detailed Description of the Invention

A general process for the preparation of compounds of Formula I is illustrated by the following equation:

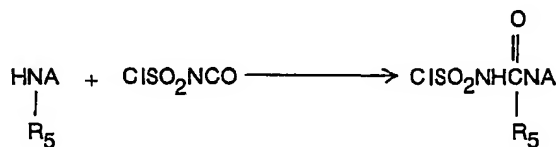


More specifically, the compounds of this invention of Formula I are prepared by contacting a heterocyclic amine of Formula V and chlorosulfonyl isocyanate to prepare the intermediate of Formula III. These

compounds are preferably generated *in situ* and used without isolation in the synthesis of compounds of Formula I. The aminocarbonyl sulfamoyl chloride of Formula III is then contacted with an indole of Formula IV, preferably in the presence of a Friedel-Crafts catalyst, to afford herbicidal indole sulfonamides of Formula I.

5

10



V

III

15

The heterocyclic amine of Formula V is dissolved or suspended in an inert organic solvent, including but not limited to, dichloromethane, nitroethane, nitropropanes, tetrahydrofuran and nitroethane. The reaction mixture is maintained in an inert atmosphere at a temperature of -80°C to 0°C . When tetrahydrofuran is the solvent, the preferred temperature range is -80°C to -50°C ; for nitromethane, the preferred range is -20° to 0° . One equivalent of chlorosulfonyl isocyanate, either neat or, if tetrahydrofuran is not the solvent, dissolved in the solvent, is contacted with the amine at such a rate as to maintain the reaction temperature within the preferred range. The reaction proceeds rapidly and the reaction mixture is maintained within the preferred temperature range for a period of 0.1 to 1.0 hour to insure complete formation of intermediate III. Compound III is then contacted at a temperature within the preferred range for the solvent employed with an equimolar amount of indole IV, either neat or dissolved in the solvent.

20

From this point on, the reaction conditions employed depend of the nature of the substituents on the pyrrole ring of the indole nucleus. For reactions with indoles IV in which R_1 is H or $\text{C}_1\text{--C}_4$ alkyl and R_2 is H or $\text{C}_1\text{--C}_3$ alkyl, a Friedel-Crafts catalyst may optionally be added at a temperature within the preferred range. Friedel-Crafts catalysts are extensively defined in Volume I, Chapter IV of "Friedel-Crafts and Related Reactions", ed. G. A. Olah, Interscience Publ., New York, 1963. Preferred catalysts are those acidic halides, typified by aluminum chloride and bromide, which possess an electron deficient central metal atom capable of electron acceptance from basic reagents. More preferred is the use of aluminum (III) chloride in catalytic quantity, the exact amount of which would be apparent to one skilled in the art. The reaction mixture is allowed to warm to ambient temperatures and is maintained under an inert atmosphere until completion, usually for a period of from 0.5 to 24 hours. Tetrahydrofuran and nitroethane are the preferred solvents for these reactions.

25

For reactions with indoles IV in which R_1 is other than H or $\text{C}_1\text{--C}_4$ alkyl or in which R_2 is $\text{SO}_2\text{C}_6\text{H}_5$, an equimolar amount of a Friedel-Crafts catalyst, preferably aluminum (III) chloride, is added to the reaction mixture at this point. The reaction is allowed to warm to ambient temperature and is then maintained under an inert atmosphere at temperatures ranging from ambient to the boiling point of the solvent employed, with nitromethane being the preferred solvent. The reaction proceeds to completion within 0.5 to 24 hours. In general, higher reaction temperatures and shorter reaction times (0.5 to 4 hours) afford the highest yield. In all cases, the intermediate III is preferably prepared in the same solvent in which the reaction with indole IV is to be carried out.

40

The compounds of the invention of Formula I may be isolated by partitioning the reaction mixture between dilute aqueous alkali and an organic solvent such as dichloromethane or chloroform. The products are soluble in the aqueous phase and may be precipitated from it after separation of the layers by the addition of a slight excess of an acid such as acetic acid or hydrochloric acid. If the products do not precipitate on acidification they may be isolated by extraction into an organic solvent such as dichloromethane, nitromethane or ethyl acetate followed by evaporation of the solvent. This procedure is most useful in cases in which tetrahydrofuran is the reaction solvent.

50

When the reaction is carried out in a water-immiscible solvent such as dichloromethane or nitromethane, isolation of the products may best be carried out by contacting the reaction mixture with water, followed by separation of the organic phase and further extraction of the products into an organic solvent such as dichloromethane, nitromethane, nitroethane or ethyl acetate. Evaporation of the organic solution affords the crude products of Formula I.

55

Purification of the reaction products may be accomplished by trituration with an appropriate solvent, recrystallization or chromatography.

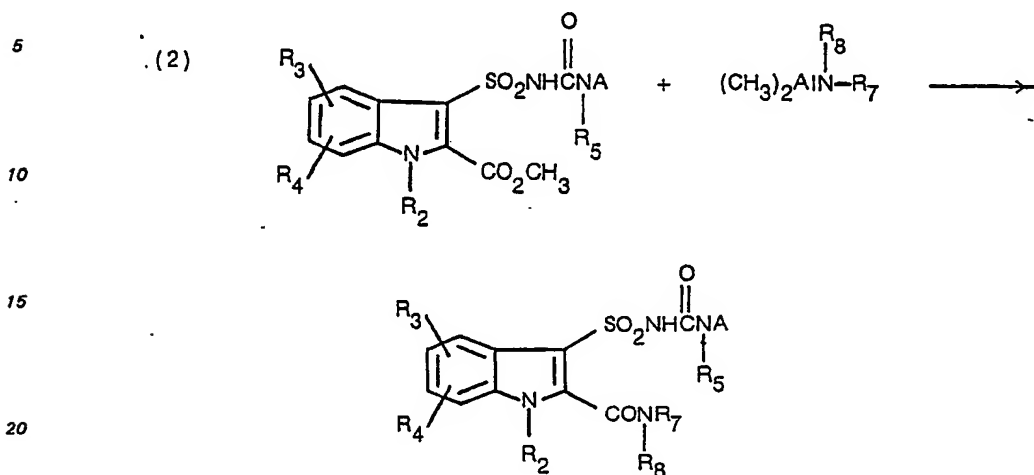
60

The procedures described generally by Equation 1 are preferred for the preparation of indole sulfonamides of Formula I in which R_1 is CO_2R_6 , $\text{C}(\text{O})\text{R}_{10}$, SO_2R_{10} or $\text{SO}_2\text{NR}_{11}\text{R}_{12}$ and R_2 is H or $\text{C}_1\text{--C}_3$ alkyl or in which R_1 is H or $\text{C}_1\text{--C}_4$ alkyl and R_2 is $\text{C}_1\text{--C}_3$ alkyl or $\text{SO}_2\text{C}_6\text{H}_5$. For cases in which R_1 is H, isomeric products of Formulae I and II ($\text{R} = \text{R}_1 = \text{H}$) are generally obtained by these methods and may be separated by column chromatography, preparative high pressure or medium pressure chromatography or similar methods.

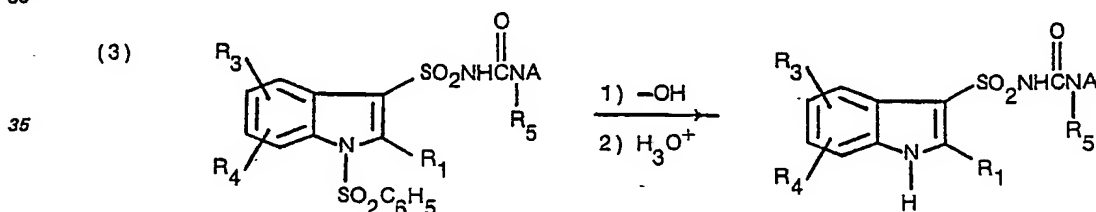
65

Indole sulfonamides of Formula I in which R_1 is $\text{C}(\text{O})\text{NR}_7\text{R}_8$ are preferably prepared from the

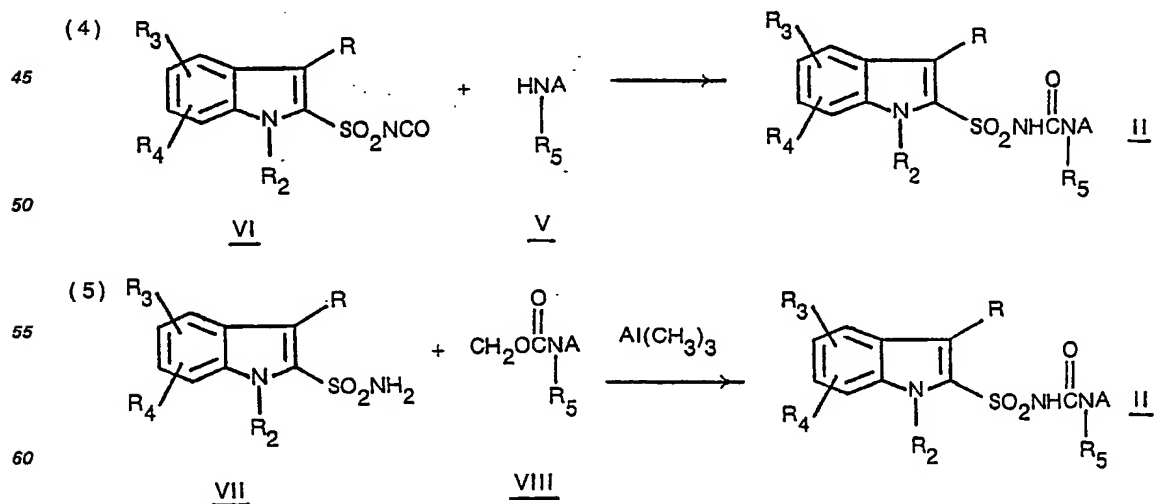
corresponding compounds in which R_1 is CO_2CH_3 by reaction with dialkylaluminum-N-alkylamide derivatives as shown in Equation 2. This method is disclosed in E.P.A. 7687, filed February 6, 1980.



Indole sulfonamides of Formula I in which R_1 is H or $\text{C}_1\text{--C}_4$ alkyl and R_2 is H are preferably prepared by alkaline hydrolysis of the corresponding compounds in which R_2 is $\text{SO}_2\text{C}_6\text{H}_5$ as shown by equation 3. The reaction is carried out in an excess of aqueous alkali, preferably in the presence of a water-miscible co-solvent such as ethanol, dioxane or tetrahydrofuran at temperatures ranging from ambient to the reflux temperature of the solvent mixture employed. After 1 to 24 hours, the reaction mixture is diluted with water and acidified, at $0\text{--}5^\circ$, with aqueous hydrochloric acid to precipitate the product.



The compounds of this invention of Formula II can be prepared by one of the two methods illustrated below in Equations 4 and 5.



As shown in Equation 4, compounds of Formula II can be prepared by reacting an amino-heterocycle V with an appropriately substituted sulfonyl isocyanate of Formula VI. The reaction is best carried out in inert, aprotic organic solvents such as acetonitrile, methylene chloride or tetrahydrofuran at ambient

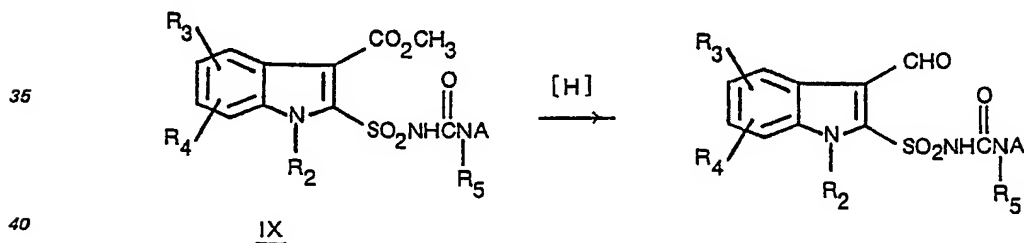
temperature. The mode of addition is not critical; however it is often convenient to add a solution of the sulfonyl isocyanate VI to a stirred solution of amino-heterocycle V. The reaction is generally exothermic. In some cases, the desired product is insoluble in the reaction medium and crystallizes from it in pure form. Products soluble in the reaction medium are isolated by evaporation of the solvent, trituration of the residue with solvents such as 1-chlorobutane, methylene chloride or ethyl ether, and filtration.

The intermediate sulfonyl isocyanates of Formula VI are prepared by reacting the corresponding sulfonamides of Formula VII with phosgene and n-butyl isocyanate at reflux in a solvent such as xylenes and in the presence of a catalytic amount of a non-nucleophilic base such as 1,4-diazabicyclo(2,2,2)octane. Useful procedures are reported by H. Ulrich and A. A. Y. Sayigh, *Newer Methods of Preparative Organic Chemistry*, Vol. VI, pp. 223—241, Academic Press, New York, W. Foerst, Ed.

Compounds of Formula II can also be prepared as shown in Equation 5 by reaction of a sulfonamide of Formula VII with an appropriate methyl carbamate of Formula VIII in the presence of trimethylaluminum. More specifically, the reaction mixture containing sulfonamide VII and an inert, aprotic solvent such as methylene chloride or toluene is reacted under an inert atmosphere with trimethylaluminum and the resulting mixture stirred at ambient temperature until gas evolution ceases. The methyl carbamate VIII is added, generally neat, and the reaction is allowed to proceed at temperatures ranging from ambient to reflux for 16 to 96 hours. The product can be isolated after addition of aqueous hydrochloric acid to the reaction mixture followed by the partitioning of the product into methylene chloride. The product may be purified by crystallization from solvents such as 1-chlorobutane, ethyl ether or methylene chloride or by chromatography.

Either of the methods shown by Equations 4 and 5 is suitable for the preparation of compounds of Formula II in which R is H, C₁—C₄ alkyl, SO₂R₁₀, SO₂NR₁₁R₁₂, CH₂N(CH₃)₂, CH₂OCH₂CH₃ or CH₂OCH₃ and R₂ is C₁—C₃ alkyl or SO₂C₆H₅. The method of Equation 4 is more preferred for preparation of Compounds II with the above listed values of R where R₂ is H. This method is also preferred for preparation of Compounds II in which R is (CH₂)_mCO₂R₉, R₉ is other than H and R₂ is H or C₁—C₃ alkyl.

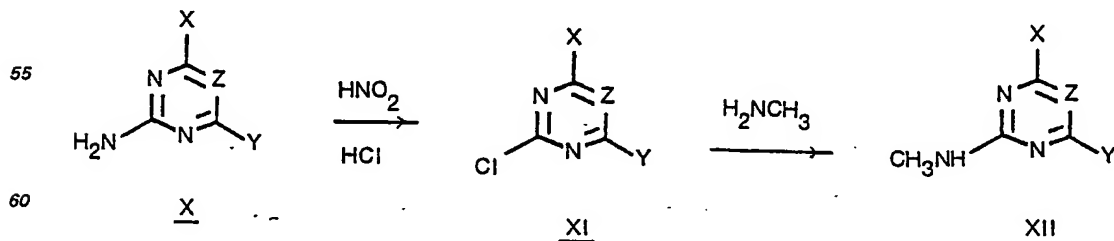
Compounds of Formula II in which R is (CH₂)_mCO₂R₉, R₉ is H and R₂ is H or C₁—C₃ alkyl may best be prepared from the corresponding compounds in which R₉ is C₁—C₃ alkyl by alkaline hydrolysis. Compounds of Formula II in which R is CHO may be prepared from the corresponding compounds in which R is (CH₂)_mCO₂R₉, m is 0 and R₉ is CH₃ (Compound IX) by partial reduction with a reducing agent such as diisobutylaluminum hydride. Procedures for these conversions would be apparent to one skilled in the art.



Preparation of Reactants

The synthesis of heterocyclic amine derivatives such as those of Formula V has been reviewed in "The Chemistry of Heterocyclic Compounds", a series published by Interscience Publ., New York and London. 2-Amino-pyrimidines are described by D. J. Brown in "The Pyrimidines", Vol. XVI of the above series. 2-Amino-1,3,5-triazines are reviewed by E. M. Smolin and L. Rapoport in "s-Triazines and Derivatives," Vol. XIII of the same series. The synthesis of triazines is also described by F. C. Schaeffer, U.S. 3,154,547 and by K. R. Huffman and F. C. Schaeffer, *J. Org. Chem.*, 28, 1812—1821 (1963).

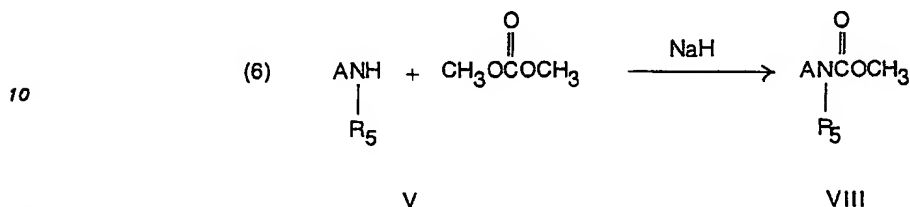
The heterocyclic amines of Formula V in which R₅ is CH₃ may be prepared by the following procedure, or by modifications thereof apparent to one skilled in the art:



A solution of the amine X in concentrated hydrochloric acid is reacted with an aqueous sodium nitrite solution and the chloro compound XI is isolated in the usual manner by filtration of the acidic solution (see for example, Bee and Rose, *J. Chem. Soc. C.*, 2051 (1966) for the case in which Z is CH and X and Y are

OCH₃). Displacement of the chlorine may be accomplished by heating with an excess of methylamine in water to obtain them ethylaminoheterocycle XII.

Pyrimidinyl and triazinyl methyl carbamates of Formule VIII may be prepared by the method illustrated in Equation 6. A heterocyclic amine of Formula V is reacted with one or two equivalents of sodium hydride, when R⁵ is CH₃ or H, respectively, and excess dimethyl carbonate to form VIII.

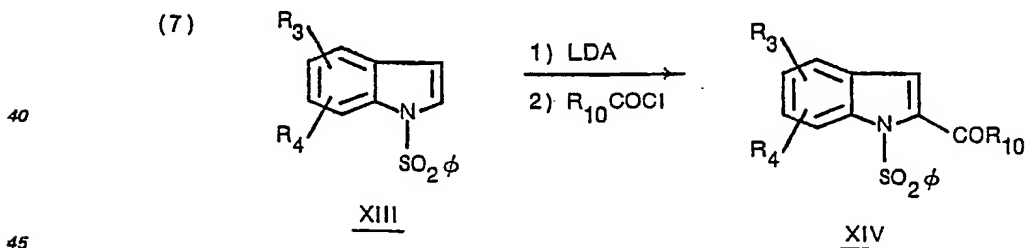


This reaction takes place in an inert solvent such as tetrahydrofuran at 25 to 70°C for 1 to 24 hours. The product is isolated by (a) addition of two equivalents of concentrated hydrochloric acid and aqueous saturated sodium chloride and (b) separation of the organic phase followed by concentration to dryness *in vacuo*.

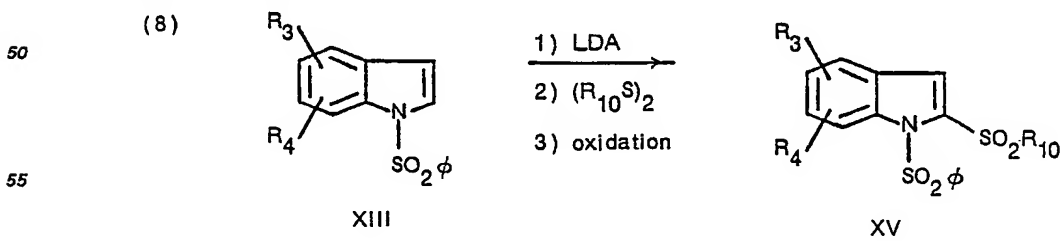
The preparation of indole derivatives has been extensively reviewed in "Indoles", Parts One-Four, Vol. XXV of the series "The Chemistry of Heterocyclic Compounds" and in "The Chemistry of Indoles", by R. J. Sundberg, Academic Press, New York and London (1970). Two other particularly useful procedures are described by P. G. Gassman in U.S. 3,901,899 and U.S. 3,960,926.

Indoles of Formula IV in which R₁ is H, C₁—C₄ alkyl or CO₂R₆ and R₂ is H or C₁—C₃ alkyl are well-known in the literature. Indoles of Formula IV in which R₁ is H or C₁—C₄ alkyl and R₂ is SO₂C₆H₅ are prepared from the corresponding compounds in which R₂ is H by reaction with one equivalent of a base, such as sodium methylsulfonylmethide or *n*-butyllithium, in ether or tetrahydrofuran, followed by reaction with phenylsulfonyl chloride (see, for example, Sundberg and Russell, *J. Org. Chem.* 38, 3324 (1973) and Saulnier and Gribble, *J. Org. Chem.* 47, 747 (1982)).

Indoles of Formula IV in which R₁ is COR₁₀, may be prepared via the method of Equation 7. Reaction of N-phenylsulfonyl indoles of Formula XIII in tetrahydrofuran with a slight excess of lithium diisopropylamine (LDA) at low temperature, followed by warming to ambient temperature, recooling and quenching the anion thus formed with an acid chloride R₁₀COCl results in the formation of compounds of Formula XIV (see, for example, Saulnier and Gribble, *ibid.*).



Indoles of Formula IV in which R₁ is SO₂R₁₀ may be prepared as illustrated by Equation 8.

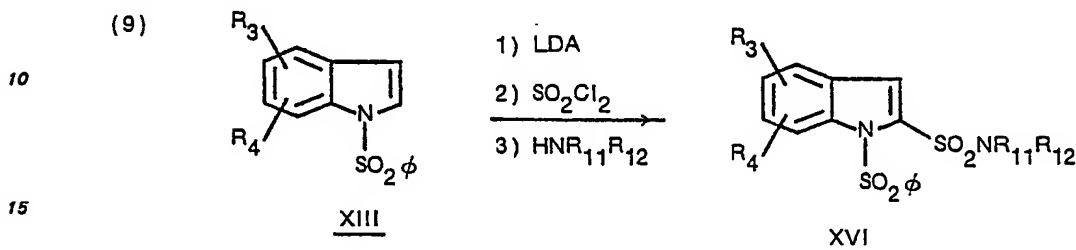


Lithiation of XIII as described above followed by quenching with a dialkyl disulfide (R₁₀S)₂ affords N-phenylsulfonyl-2-alkylthioindoles, which may be oxidized to alkyl sulfonyl indoles of Formule XV by methods described by Wieland, et. al., *Justus Liebigs Ann. Chem.* 713, 186 (1968) and Hino, et. al., *J. Chem. Soc., Sect. D* 473 (1972).

Equation (9) represents a method of preparation for indoles of Formula IV in which R₁ is SO₂NR₁₁R₁₂. Lithiation in this method is carried out under an inert atmosphere by the slow addition of an ether solution of XIII to 1.1 equivalents of LDA at 0° followed by stirring at 0° for 0.5 to 2 hours. The resulting slurry is

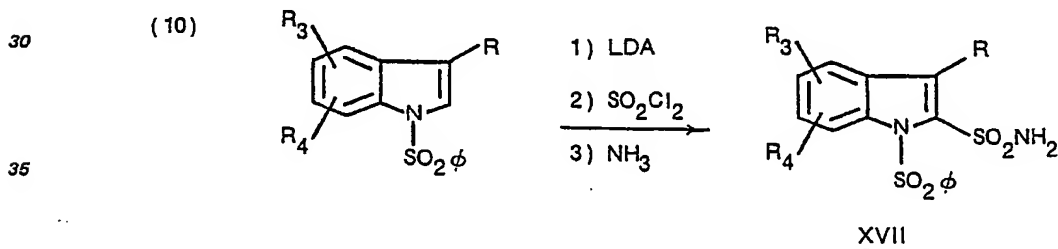
added to a solution of 2 equivalents of sulfonyl chloride in an equal volume of hexanes at -20° to -30° . After 0.5 to 1 hour at -20° to -30° , the reaction mixture is allowed to warm to ambient temperature, stirred until completion (generally 2 to 8 hours), then diluted with water. The N-phenylsulfonyl-2-chlorosulfonyl indoles are isolated by extraction into ethyl acetate and purified by trituration with ether or similar solvents.

- 5 Reaction of these products with an excess of amine $\text{HNR}_{11}\text{R}_{12}$ using procedures that would be obvious to one skilled in the art affords indole sulfonamides XVI.

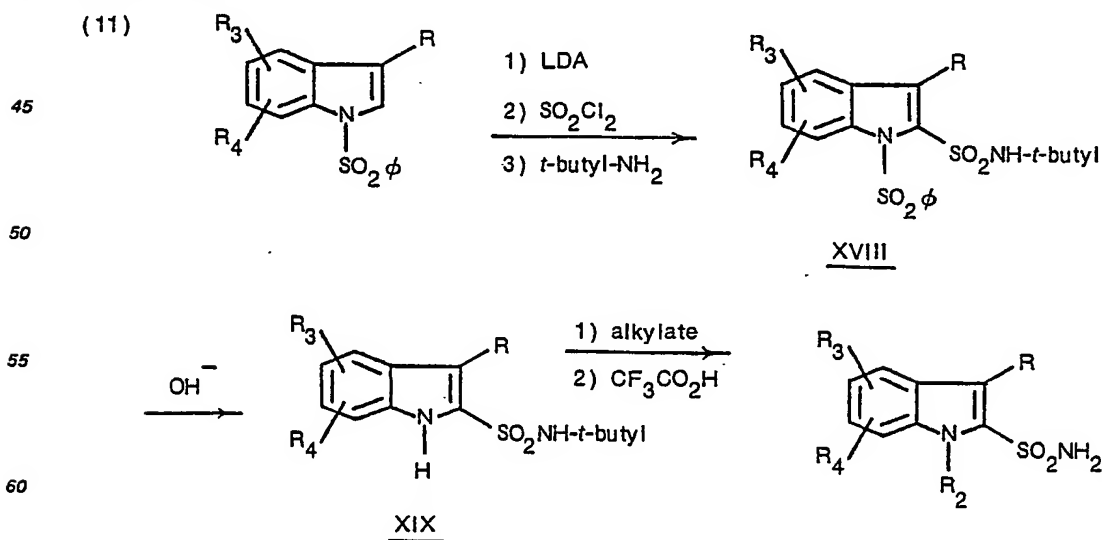


Indoles of Formula IV in which R_1 is COR_{10} , SO_2R_{10} or $\text{SO}_2\text{NR}_{11}\text{R}_{12}$ and R_2 is H are prepared from the corresponding indoles in which R_2 is $\text{SO}_2\text{C}_6\text{H}_5$ (compounds XIV, XV and XVI respectively) by alkaline hydrolysis, according to the procedures of Equation 3. In cases in which the product does not precipitate upon acidification, it may be isolated by extraction into ethyl acetate or ether. Alkylation of these indoles or nitrogen by procedures well-known in the literature affords compounds of Formula IV in which R_1 is COR_{10} , SO_2R_{10} or $\text{SO}_2\text{NR}_{11}\text{R}_{12}$ and R_2 is $\text{C}_1\text{--C}_3$ alkyl.

Indole-2-sulfonamides of Formula VII in which R is H, $\text{C}_1\text{--C}_4$ alkyl, CH_2OCH_3 or $\text{CH}_2\text{OCH}_2\text{CH}_3$ and R_2 is $\text{SO}_2\text{C}_6\text{H}_5$ may be prepared by the method of Equation 10 according to the procedures of Equation 9. The corresponding compounds of Formula VII in which R_2 is H may be prepared by alkaline hydrolysis in XVII according to the procedures of Equation 3.



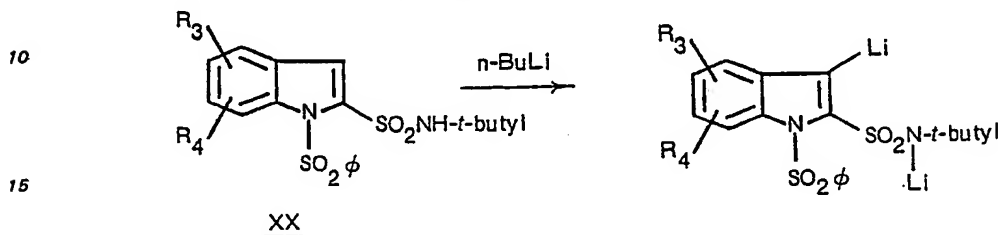
Preparation of the corresponding compounds in which R_2 is $\text{C}_1\text{--C}_3$ alkyl may be carried out by the method illustrated below.



The protected indole-2-sulfonamide of Formula XVIII in which R is H, $\text{C}_1\text{--C}_4$ alkyl, CH_2OCH_3 or $\text{CH}_2\text{OCH}_2\text{CH}_3$ is prepared by the method of Equation 9. Alkaline hydrolysis as described above affords XIX which may be

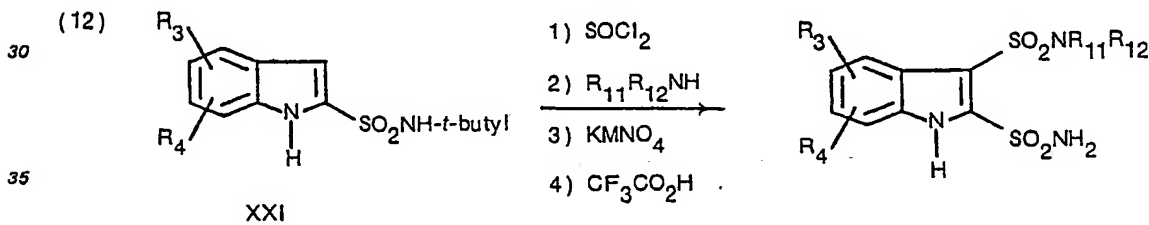
alkylated on the more nucleophilic indole nitrogen after dianion formation by procedures which would be known to one skilled in the art. The t-butyl protecting group is then removed with trifluoroacetic acid to afford the desired indole-2-sulfonamides in which R_2 is C_1-C_3 alkyl.

Lithiation of protected indole-2-sulfonamides of Formula XVIII in which R is H (compound XX) provides access to compound of Formula VII in which R is SO_2R_{10} or $(CH_2)_mCO_2R_9$ and m is 0 or 1. Reaction of indole XX under an inert atmosphere with 2 equivalents of n-butyl lithium in ether or tetrahydrofuran at -20° to 0° , optionally in the presence of tetraethylenediamine, affords the 3-lithio derivative.



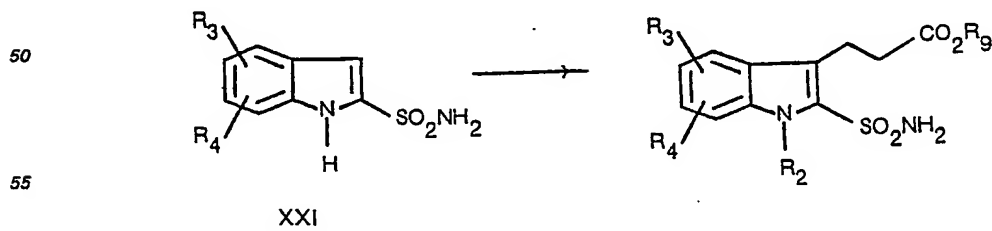
Quenching this anion with a C_1-C_3 alkyl chloroformate, a C_1-C_3 alkyl α bromoacetate or a C_1-C_3 dialkyl disulfide, followed in the last case by oxidation, affords indole-2-sulfonamides of Formula XVIII in which R is $(CH_2)_0CO_2R_9$, $(CH_2)_1CO_2R_9$ or SO_2R_{10} respectively. Suitable procedures for these conversions would be known to one skilled in the art. Indole-2-sulfonamides of Formula VII in which R_2 is H or C_1-C_3 alkyl, R is SO_2R_{10} or $(CH_2)_mCO_2R_9$ and m is 0 or 1 may then be prepared, using procedures already described or well-known in the literature, by alkaline hydrolysis of the N-phenylsulfonyl group, re-esterification when R is $(CH_2)_mCO_2R_9$, N-alkylation if desired, and removal of the t-butyl protecting group.

Indoles of Formula VII in which R is $SO_2NR_{11}R_{12}$ are prepared from the corresponding compounds of Formula XXI, obtained by alkaline hydrolysis of XX, by the procedure shown in Equation 12.



Reaction of XXI with thionyl chloride affords the indole-3-sulfonyl chloride, which is converted to the sulfonamide and oxidized to the sulfonamide using procedures described by J. Szmuszkovicz in Canadian Patent 747,920 and in *J. Org. Chem.* 29, 179 (1964). Optional alkylation of the indole nitrogen followed by trifluoroacetic acid hydrolysis as described above afford indoles VII in which R is $SO_2NR_{11}R_{12}$ and R_2 is H or C_1-C_3 alkyl.

Reaction of indole XXI with acrylic acid in the presence of acetic acid affords the indole-3-propionic acid. Esterification, alkylation of the indole nitrogen, if desired, and trifluoroacetic acid hydrolysis gives rise to indoles of Formula VII in which R is $(CH_2)_mCO_2R_9$, m is 2 and R_2 is H or C_1-C_3 alkyl.



Reaction of indole XXI with formaldehyde and dimethylamine under Mannich reaction conditions followed by optional alkylation of the indole nitrogen and removal of the t-butyl protecting group affords indoles of Formula VII in which R is $CH_2N(CH_3)_2$ and R_2 is H or C_1-C_3 alkyl.

Agriculturally suitable salts of Formula I and II are also useful herbicides and can be prepared by a number of ways known to the art.

The compounds of the invention and their preparation are further illustrated by the following samples. Unless otherwise indicated, all parts are by weight and all temperatures are in $^\circ C$.

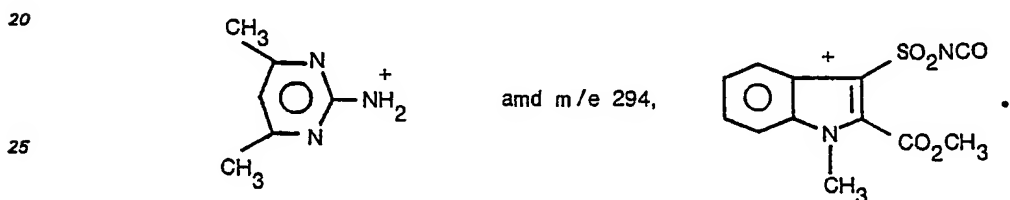
0 070 698

Example 1

Preparation of Methyl 3-[[[4,6-dimethylpyrimidin-2-yl]aminocarbonyl]aminosulfonyl]-1-methyl-1H-indole-2-carboxylate.

To a stirred suspension of 2.59 g (0.021 mole) of 2-amino-4,6-dimethylpyrimidine in 75 ml dry nitromethane at -10° was added dropwise under nitrogen via syringe 2.0 ml (0.023 mole) of chlorosulfonyl isocyanate at such a rate as to maintain the temperature below 0° . The resulting clear solution was stirred for 0.5 hour at -5° to -10° and then contacted dropwise with a solution of 3.97 g (0.021 mole) of methyl 1-methyl-1H-indole-2-carboxylate dissolved in 40 ml of dry nitromethane. Upon completion of the addition, 2.95 g (0.022 mole) of aluminum (III) chloride was added in one portion. The reaction mixture was stirred at reflux for 3 hours, then cooled to room temperature and poured into 300 ml H_2O . Methylene chloride was added and the layers separated. The aqueous solution was extracted with two additional portions of CH_2Cl_2 and the combined organic solutions were washed with H_2O , dried over $MgSO_4$ and evaporated *in vacuo*. Trituration of the residue with minimal CH_2Cl_2 afforded 2.02 g of the desired product as a white solid, m.p. $212-215^{\circ}$ (d).

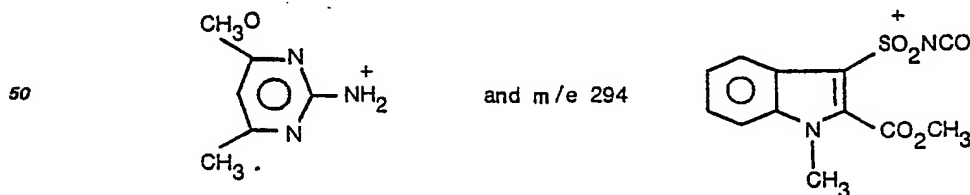
The infrared spectrum of the product included absorptions at 3215 and 3150 (NH), 1730 (C=O), 1710 (C=O), 1345 and 1150 (SO_2) cm^{-1} , 1H NMR ($CDCl_3/DMSO-d_6$) δ 2.43 (s, 6H, pyrimidine CH_3), 3.88 (s, 6H, ester CH_3 and N- CH_3), 6.90 (s, 1H, pyrimidine CH), 7.23-7.75 (m, 3H, indole CH), 8.16-8.47 (m, 1H, indole CH), 10.30 (s, 1H, NH), 13.26 (br s, 1H, NH). Mass spectral analysis showed m/e 123.



Example 2

Preparation Methyl 3-[[[4-methoxy-6-methylpyrimidin-2-yl]aminocarbonyl]aminosulfonyl]-1-methyl-1H-indole-2-carboxylate.

To a stirred suspension of 2.92 g (0.021 mole) of 2-amino-4-methoxy-6-methylpyrimidine in 75 ml dry CH_3NO_2 at -10° was added dropwise under nitrogen via syringe 2.0 ml (0.023 mole) of chlorosulfonyl isocyanate at such a rate as to maintain the temperature below 0° . The resulting clear solution was stirred 0.5 hour at -5° to -10° and then contacted dropwise with a solution of 3.97 g (0.021 mole) of methyl 1-methyl-1H-indole-2-carboxylate dissolved in 25 ml of dry nitromethane. Upon completion of the addition, 2.95 g (0.022 mole) of aluminum (III) chloride was added in one portion. The reaction mixture was stirred at reflux for 3 hours, then cooled to room temperature and poured into 300 ml H_2O . Dichloromethane was added and the layers were separated. The aqueous solution was extracted with two additional portions of CH_2Cl_2 and the combined organic solutions washed with H_2O , dried over $MgSO_4$ and evaporated *in vacuo*. Chromatography on silica gel with 5% acetone in CH_2Cl_2 afforded 1.63 g of the desired product as a white solid, m.p. $200-203^{\circ}$ (d). The infrared spectrum of the product included absorptions at 3190 (NH), 1715 (C=O), 1703 (C=O), 1340 and 1150 (SO_2) cm^{-1} , 1H NMR ($CDCl_3/DMSO-d_6$) δ 2.45 (s, 3H, pyrimidine CH_3), 3.95 (s, 3H, OCH_3), 4.0 (s, 6H, OCH_3 and N- CH_3), 6.37 (s, 1H, pyrimidine CH), 7.23-7.56 (m, 3H, indole CH), 8.18-8.47 (m, 1H, indole CH), 9.50 (s, 1H, NH), 13.08 (br s, 1H, NH). Mass spectral analysis showed m/e 139.



Example 3

Preparation of 1-phenylsulfonyl-1H-indole-2-sulfonyl chloride

To a stirred solution of lithium diisopropylamine (prepared from .124 mole diisopropylamine in 50 ml ether and 82 ml of 1.6 M n-butyllithium in hexanes) at 0° was added dropwise under nitrogen over 40 minutes a solution of 29.2 g (.1135 mole) of 1-phenylsulfonyl-1H-indole in 400 ml dry ether. The resulting slurry was stirred 30 minutes at 0° , then transferred via canula to a solution of 18.3 ml (.227 mole) of sulfonyl chloride in 400 ml hexanes at -20° to -30° (dry ice/ CCl_4 bath). Near the end of the addition there was a gradual 5° exotherm. After 30 min. additional cooling, the ice bath was removed and stirring continued for 4 hours at ambient temperature. The slurry was poured into water and ethyl acetate was added with stirring until all solids dissolved. The layers were separated and the aqueous phase extracted 2 times with ethyl

acetate. The combined organic solutions were washed with water, dried over MgSO_4 and evaporated *in vacuo* to a dark semisolid. Ether trituration afforded 25.07 g of tan solid, m.p. 118—120° and a second crop of 3.60 g tan solid, m.p. 115—118°. ^1H NMR (CDCl_3) δ 7.17—7.84 (m, 7H), 8.03—8.21 (m, 2H), 8.36 (d, 1H, $J=9\text{Hz}$, C—3 proton). The infrared spectrum of the product included absorptions at 1385, 1180 and 1185 cm^{-1} for sulfonyl chloride.

Example 4

Preparation of 1-phenylsulfonyl-1H-indole-2-sulfonamide.

To a stirred solution of 10 g (.0281 mole) of 1-phenylsulfonyl-1H-indole-2-sulfonyl chloride in 150 ml anhydrous THF was added at -78° 4 ml (.192 mole) of anhydrous ammonia. The resulting slurry was allowed to warm to room temperature over 30 minutes, then was sparged with nitrogen to remove excess ammonia. The slurry was filtered and the filtrate evaporated *in vacuo* to afford 9.19 g of tan solid, m.p. 199—200.5°. ^1H NMR ($\text{CDCl}_3/\text{DMSO}-d_6$) δ 7.23—7.77 (m, 9H), 8.1—8.28 (m, 3H, C—3 proton and SO_2NH_2). The infrared spectrum of the product included absorptions at 3400 and 3280 cm^{-1} (SO_2NH_2).

Example 5

Preparation of 1H-indole-2-sulfonamide.

To a stirred solution of 2.4 g (7.13 mmole) of 1-phenylsulfonyl-1H-indole-2-sulfonamide in 50 ml THF was added 0.86 g (21 mmol) of sodium hydroxide dissolved in 5 ml H_2O . The mixture was refluxed for 5 hours, then cooled to room temperature, poured into water and acidified slightly with 1N HCl. This solution was extracted 2 times with ethyl acetate and the combined organic layers were dried over MgSO_4 and evaporated *in vacuo* to afford 0.58 g of a pale tan solid, m.p. 190—192°. ^1H NMR ($\text{CDCl}_3/\text{DMSO}-d_6$) δ 6.92—7.72 (m, 7H), 11.59 (br, 1H, indole NH). The infrared spectrum of the product included absorptions at 3385, 3280, 1310 and 1140 (SO_2NH_2) and at 3340 cm^{-1} (indole NH).

Example 6

Preparation of N-[(4,6-dimethylpyrimidin-2-yl)aminocarbonyl]-1H-indole-2-sulfonamide.

A mixture of 5.0 g (.0255 mole) of 1H-indole-2-sulfonamide, 2.9 ml (.0255 mole) of n-butylisocyanate, a catalytic amount of 1,4-diazabicyclo[2,2,2] octane and 75 ml of xylenes was heated under nitrogen to 138°. Phosgene was condensed into the reaction mixture until the temperature fell to 130°. As phosgene was consumed, the temperature gradually increased to 138°.

Further additions of phosgene were made until the reaction temperature failed to return to 138°. Unreacted phosgene was removed with a nitrogen stream (2N NaOH trap) and heating was continued for 15 minutes. The reaction mixture was cooled to room temperature, filtered under nitrogen and evaporated *in vacuo* to afford the sulfonylisocyanate intermediate which was used without purification in the second step of the reaction.

A solution of 1.0 g (4.47 mmol) of the above isocyanate in 5 ml dry acetonitrile was added under nitrogen to a mixture of 0.5 g (4.06 mmol) of 2-amino-4,6-dimethylpyrimidine in 5 ml dry acetonitrile and the mixture allowed to stir overnight at ambient temperature. The precipitate was collected by filtration and washed with ether to afford the desired material.

Example 7

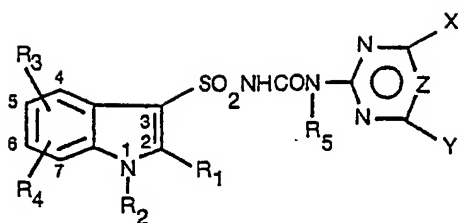
Preparation of N-[(4,6-dimethylpyrimidin-2-yl)aminocarbonyl]-1-phenylsulfonyl-1H-indole-2-sulfonamide.

To a slurry of 1.0 g (3.0 mmol) of 1-phenylsulfonyl-1H-indole-2-sulfonamide in 20 ml methylene chloride was added under nitrogen at ambient temperature 1.65 ml (3.3 mmol) of a 2M trimethylaluminum solution in toluene. The resulting mixture was stirred until gas evolution had ceased, then 0.54 g (3 mmol) of methyl (4,6-dimethylpyrimidin-2-yl) carbamate was added in one portion. The mixture was stirred at reflux overnight, then cooled to room temperature, poured into 50 ml of ice-cold 5% HCl and stirred 5 minutes. The layers were separated and the aqueous phase extracted 2 times with ethyl acetate. The combined organic layers were washed with H_2O , dried over MgSO_4 and evaporated *in vacuo* to afford the desired product.

By using methods described generally above and illustrated in Examples 1 through 7, compounds as shown in the following tables can similarly be prepared. These tables are not meant to be all inclusive but only illustrative of the breadth of the invention.

0 070 698

TABLE I



R ₁	R ₂	R ₃	R ₄	R ₅	X	Y	Z	m.p. (°C)
H	H	H	H	H	CH ₃	CH ₃	CH	
H	H	H	H	H	CH ₃	OCH ₃	CH	
H	CH ₃	H	H	H	CH ₃	OCH ₃	N	
H	C ₃ H ₇	H	H	CH ₃	OCH ₃	OCH ₃	CH	
H	C ₂ H ₅	5-NO ₂	H	H	CH ₃	CH ₂ OCH ₃	N	
H	H	H	6-Cl	H	OCH ₃	Cl	CH	
H	H	H	H	H	OCH ₃	N(CH ₃) ₂	CH	
H	SO ₂ C ₆ H ₅	H	H	H	CH ₃	CH ₃	CH	
H	SO ₂ C ₆ H ₅	H	H	H	OCH ₃	CH ₃	N	
H	SO ₂ C ₆ H ₅	5-Cl	7-Cl	H	CH ₃	OCH ₂ CH ₃	CH	
H	SO ₂ C ₆ H ₅	6-CH ₃	H	CH ₃	CH ₃	H	CH	
CH ₃	H	H	H	H	OCH ₃	CH ₃	CH	
CH ₃	H	H	H	H	OCH ₃	CH ₃	N	
CH ₃	H	H	H	H	CH ₃	CH ₃	N	
CH ₃	H	H	H	H	CH ₃	CH ₃	CH	
CH ₃	H	H	H	CH ₃	OCH ₃	OCH ₃	CH	
CH ₃	CH ₃	H	H	H	OCH ₃	CH ₃	N	
CH ₃	CH ₃	H	H	H	OCH ₃	CH ₃	CH	
CH ₃	CH ₃	H	H	H	CH ₃	CH ₃	CH	
CH ₃	CH ₃	H	H	H	OCH ₃	OCH ₃	CH	
CH ₃	C ₂ H ₅	5-Cl	H	H	CH ₃	CH ₃	N	
C ₂ H ₅	H	6-OCH ₃	H	H	OCH ₃	CH ₃	CH	
C ₂ H ₅	C ₂ H ₅	H	H	H	OCH ₃	OCH ₃	N	
C ₂ H ₅	H	H	H	H	CH ₃	CH ₂ OCH ₃	CH	




























0 070 698

TABLE I (Continued)

R ₁	R ₂	R ₃	R ₄	R ₅	X	Y	Z	m.p. (°C)
n-C ₄ H ₉	H	H	H	H	CH ₃	CH ₃	N	
s-C ₄ H ₉	H	7-C ₂ H ₅	H	H	CH ₃	OCH ₃	N	
i-C ₃ H ₇	CH ₃	H	H	H	OCH ₃	CH ₃	CH	
i-C ₃ H ₇	CH ₃	H	H	H	OCH ₃	CH ₃	N	
i-C ₃ H ₇	CH ₃	H	H	H	OCH ₃	OCH ₃	CH	
i-C ₃ H ₇	CH ₃	H	H	H	CH ₃	CH ₃	CH	
i-C ₃ H ₇	H	H	H	H	CH ₃	CH ₃	N	
i-C ₃ H ₇	H	H	H	CH ₃	OCH ₃	CH ₃	N	
i-C ₃ H ₇	H	H	H	H	OCH ₃	CH ₃	CH	
i-C ₃ H ₇	H	4-OC ₃ H ₇	H	H	CH ₃	CH ₃	CH	
i-C ₃ H ₇	H	H	H	H	OCH ₃	OCH ₃	CH	
i-C ₃ H ₇	CH ₃	H	H	H	CH ₃	CH ₃	N	
i-C ₃ H ₇	i-C ₃ H ₇	H	7-Br	H	CH ₃	CH ₂ OCH ₃	N	
CO ₂ CH ₃	CH ₃	H	H	H	CH ₃	CH ₃	CH	212-215(d)
CO ₂ CH ₃	C ₂ H ₅	7-Cl	5-Cl	H	CH ₃	CH ₂ OCH ₃	CH	
CO ₂ CH ₃	C ₂ H ₅	H	H	H	CH ₃	CH ₃	CH	
CO ₂ CH ₃	C ₃ H ₇	H	H	H	OCH ₃	CH ₃	N	
CO ₂ CH ₃	C ₃ H ₇	H	H	H	OCH ₃	CH ₃	N	
CO ₂ C ₂ H ₅	H	H	H	H	OCH ₃	CH ₃	N	205-207
CO ₂ C ₂ H ₅	H	H	H	H	OCH ₃	CH ₃	CH	143-145(d)
CO ₂ C ₂ H ₅	H	H	H	H	CH ₃	CH ₃	CH	134-138(d)
CO ₂ C ₂ H ₅	H	H	H	H	CH ₃	CH ₃	N	
CO ₂ C ₂ H ₅	H	H	H	H	OCH ₃	OCH ₃	N	165-169(d)
CO ₂ C ₂ H ₅	H	H	H	H	OCH ₃	OCH ₃	CH	197-201
CO ₂ C ₂ H ₅	H	H	H	H	CH ₃	N(CH ₃) ₂	CH	
CO ₂ C ₂ H ₅	H	5-OC ₂ H ₅	H	H	OCH ₃	OCH ₃	N	
CO ₂ C ₂ H ₅	H	H	H	CH ₃	OCH ₃	OCH ₃	CH	
CO ₂ C ₂ H ₅	H	5-C ₂ H ₅	H	H	CH ₃	CH ₃	CH	123-217(d)
CO ₂ C ₂ H ₅	CH ₃	H	4-Br	H	CH ₃	OCH ₃	N	

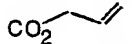
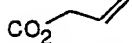
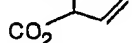
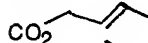

0 070 698

TABLE I (Continued)

R ₁	R ₂	R ₃	R ₄	R ₅	X	Y	Z	m.p. (°C)
CO ₂ C ₂ H ₅	CH ₃	H	H	H	CH ₃	OCH ₃	CH	
CO ₂ C ₂ H ₅	CH ₃	H	H	H	CH ₃	CH ₂ OCH ₃	CH	
CO ₂ — 	H	4-F	H	H	OCH ₃	OCH ₃	N	
CO ₂ — 	H	H	H	H	OCH ₃	OCH ₃	CH	
CO ₂ — 	H	H	H	H	OCH ₃	CH ₃	CH	
CO ₂ — 	H	6-OC ₂ H ₅	H	H	OCH ₃	CH ₃	N	
CO ₂ — 	H	H	H	H	CH ₃	CH ₃	CH	
CO ₂ — 	H	6-Br	H	H	CH ₃	CH ₃	N	
CO ₂ — 	CH ₃	H	H	H	CH ₃	OCH ₃	N	
CO ₂ — 	H	H	H	H	CH ₃	OCH ₃	CH	
CO ₂ — 	H	H	H	H	CH ₃	CH ₃	CH	
CO ₂ — 	CH ₃	H	6-Br	CH ₃	CH ₃	CH ₃	N	
CO ₂ — 	CH ₃	H	H	H	OCH ₃	OCH ₃	CH	
CO ₂ — 	CH ₃	H	H	H	OCH ₃	OCH ₃	N	
CO ₂ — 	C ₂ H ₅	5-CH ₃	H	H	OCH ₃	OC ₂ H ₅	N	
CO ₂ — 	i-C ₃ H ₇	7-C ₃ H ₇	H	H	OCH ₃	H	N	
CO ₂ — 	H	H	H	H	OCH ₃	OCH ₃	N	
CO ₂ — 	H	H	H	H	OCH ₃	OCH ₃	CH	
CO ₂ — 	H	H	H	H	OCH ₃	CH ₃	N	
CO ₂ — 	H	H	H	H	OCH ₃	CH ₃	CH	
CO ₂ — 	H	5-Br	H	CH ₃	CH ₃	CH ₂ CH ₃	N	
CO ₂ — 	H	H	H	H	CH ₃	CH ₃	CH	
CO ₂ — 	CH ₃	H	H	H	CH ₃	OCH ₃	N	
CO ₂ — 	CH ₃	H	H	H	CH ₃	OCH ₃	CH	
CO ₂ — 	CH ₃	H	H	H	CH ₃	CH ₃	N	
CO ₂ — 	CH ₃	H	H	H	CH ₃	CH ₃	CH	
CO ₂ — 	CH ₃	H	H	H	OCH ₃	OCH ₃	CH	
CO ₂ — 	CH ₃	H	H	H	OCH ₃	OCH ₂ CH ₃	N	
CO ₂ — 	C ₃ H ₇	H	H	H	OCH ₃	OCH ₃	N	

O 070 698

TABLE I (Continued)

R ₁	R ₂	R ₃	R ₄	R ₅	X	Y	Z	m.p. (°C)
	i-C ₃ H ₇	H	H	CH ₃	OCH ₃	Cl	CH	
	C ₂ H ₅	H	H	H	OCH ₃	CH ₃	N	
	H	4-OCH ₃	H	H	OCH ₃	CH ₂ CH ₃	CH	
	H	6-NO ₂	H	H	CH ₃	CH ₃	CH	
	CH ₃	H	H	H	CH ₃	CH ₂ OCH ₃	CH	
CO ₂ -t-C ₄ H ₉	H	H	H	H	CH ₃	OCH ₃	N	
CO ₂ n-C ₄ H ₉	H	H	H	H	OCH ₃	OCH ₃	CH	
CO ₂ s-C ₄ H ₉	H	H	H	H	OCH ₃	CH ₃	CH	
CO ₂ (CH ₂) ₂ Cl	H	H	H	H	OCH ₃	CH ₃	N	
CO ₂ (CH ₂) ₂ Cl	H	4-C ₂ H ₅	H	H	OCH ₃	CH ₃	CH	
CO ₂ (CH ₂) ₂ Cl	CH ₃	H	H	H	CH ₃	OC ₂ H ₅	CH	
CO ₂ (CH ₂) ₂ Cl	H	H	H	CH ₃	CH ₃	OCH ₃	CH	
CO ₂ (CH ₂) ₂ Cl	H	H	H	H	CH ₃	CH ₃	CH	
CO ₂ (CH ₂) ₂ OCH ₃	H	H	H	H	CH ₃	Cl	CH	
CO ₂ (CH ₂) ₂ OCH ₃	H	H	H	H	OCH ₃	CH ₂ OCH ₃	N	
CO ₂ (CH ₂) ₂ OCH ₃	H	7-OC ₃ H ₇	H	H	OCH ₃	H	CH	
CO ₂ (CH ₂) ₂ OCH ₃	i-C ₃ H ₇	H	H	H	OCH ₃	CH ₃	CH	
CO ₂ (CH ₂) ₂ OCH ₃	H	H	H	H	OCH ₃	CH ₃	CH	
CO ₂ (CH ₂) ₂ OCH ₃	H	H	H	H	OCH ₃	N(CH ₃) ₂	N	
CON(CH ₃) ₂	H	H	6-Br	H	OCH ₃	OCH ₃	N	
CON(CH ₃) ₂	H	7-F	H	H	CH ₃	CH ₃	N	
CON(CH ₃) ₂	H	H	H	H	CH ₃	CH ₃	CH	
CON(CH ₃) ₂	H	H	H	H	CH ₃	OCH ₃	CH	
CON(CH ₃) ₂	H	H	H	H	CH ₃	OCH ₃	N	
CON(CH ₃) ₂	H	H	H	CH ₃	OCH ₃	OCH ₃	CH	
CON(CH ₃) ₂	CH ₃	H	H	H	OCH ₃	CH ₃	N	
CON(CH ₃) ₂	CH ₃	H	H	H	OCH ₃	H	CH	
CON(CH ₃) ₂	CH ₃	H	H	H	CH ₃	CH ₃	CH	
CON(CH ₃) ₂	CH ₃	6-OCH ₃	H	H	OCH ₃	OCH ₃	CH	

0 070 698

TABLE I (Continued)

R ₁	R ₂	R ₃	R ₄	R ₅	X	Y	Z	m.p. (°C)
CON(CH ₃) ₂	CH ₃	H	H	H	OCH ₃	OCH ₃	N	
CON(CH ₃) ₂	CH ₃	H	H	H	CH ₃	CH ₃	N	
CON(CH ₃) ₂	C ₂ H ₅	H	H	H	CH ₃	OCH ₂ CH ₃	N	
CON(CH ₃) ₂	i-C ₃ H ₇	H	H	H	CH ₃	OCH ₃	N	
CON(CH ₃) ₂	C ₃ H ₇	H	H	H	CH ₃	OCH ₃	CH	
CONHCH ₃	H	H	H	H	CH ₃	N(CH ₃) ₂	CH	
CONHCH ₃	H	H	7-Cl	CH ₃	OCH ₃	CH ₃	CH	
CONHC ₂ H ₅	H	H	H	H	OCH ₃	CH ₃	N	
CONHC ₂ H ₅	CH ₃	H	H	H	OCH ₃	CH ₂ OCH ₃	CH	
CON(CH ₃)C ₂ H ₅	CH ₃	H	H	H	OCH ₃	OCH ₃	N	
CON(CH ₃)C ₂ H ₅	H	5-F	H	H	OCH ₃	CH ₂ CH ₃	N	
CONHC ₃ H ₇	H	H	H	H	CH ₃	CH ₃	CH	
CONH-i-C ₄ H ₉	H	5-OC ₃ H ₇	H	H	OCH ₃	CH ₃	N	
CON(CH ₃)C ₃ H ₇	H	H	H	H	CH ₃	CH ₃	N	
CONH-i-C ₄ H ₉	H	H	H	CH ₃	OCH ₃	CH ₃	CH	
CONH-i-C ₃ H ₇	H	H	H	H	CH ₃	OCH ₃	N	
COCH ₃	H	H	H	H	OCH ₃	OCH ₃	CH	
COCH ₃	CH ₃	H	H	H	CH ₃	CH ₃	N	
COC ₂ H ₅	H	H	H	CH ₃	CH ₃	OCH ₃	N	
COC ₂ H ₅	H	H	H	H	CH ₃	CH ₃	CH	
COC ₂ H ₅	H	H	H	H	CH ₃	CH ₂ OCH ₃	CH	
COC ₃ H ₇	H	6-NO ₂	H	H	CH ₃	OCH ₃	N	
COC ₃ H ₇	i-C ₃ H ₇	4-Br	H	H	OCH ₃	CH ₃	N	
COC ₃ H ₇	H	H	H	H	OCH ₃	CH ₃	N	
CO-i-C ₃ H ₇	H	H	H	H	OCH ₃	CH ₃	CH	
CO-i-C ₃ H ₇	H	H	H	H	OCH ₃	OCH ₃	N	
SO ₂ CH ₃	H	H	H	H	OCH ₃	OCH ₃	N	
SO ₂ CH ₃	H	H	H	H	OCH ₃	OCH ₃	CH	
SO ₂ CH ₃	H	7-OC ₂ H ₅	H	CH ₃	CH ₃	OCH ₃	N	

0 070 698

TABLE I (Continued)

R ₁	R ₂	R ₃	R ₄	R ₅	X	Y	Z	m.p. (°C)
SO ₂ CH ₃	H	H	H	H	CH ₃	OCH ₃	CH	
SO ₂ CH ₃	H	H	H	H	CH ₃	OCH ₃	N	
SO ₂ CH ₃	H	H	H	H	CH ₃	CH ₃	CH	
SO ₂ CH ₃	CH ₃	H	H	H	CH ₃	CH ₃	CH	
SO ₂ CH ₃	CH ₃	H	H	H	CH ₃	CH ₃	N	
SO ₂ CH ₃	CH ₃	5-OCH ₃	H	H	CH ₃	OCH ₃	N	
SO ₂ CH ₃	CH ₃	H	H	H	CH ₃	OCH ₃	CH	
SO ₂ CH ₃	CH ₃	H	H	H	OCH ₃	OCH ₃	CH	
SO ₂ CH ₃	CH ₃	H	H	H	OCH ₃	OCH ₃	N	
SO ₂ CH ₃	H	H	H	H	CH ₃	C ₂ H ₅	CH	
SO ₂ CH ₃	H	H	H	H	CH ₃	OC ₂ H ₅	CH	
SO ₂ CH ₃	H	4-Cl	6-Cl	H	CH ₃	Cl	CH	
SO ₂ CH ₃	CH ₃	4-Cl	H	H	CH ₃	N(CH ₃) ₂	CH	
SO ₂ CH ₃	CH ₃	4-Cl	H	H	OCH ₃	OCH ₃	CH	
SO ₂ CH ₃	CH ₃	4-Cl	H	H	OCH ₃	Cl	CH	
SO ₂ C ₂ H ₅	H	H	H	CH ₃	OCH ₃	OCH ₃	N	
SO ₂ C ₂ H ₅	H	H	6-Br	H	OCH ₃	OCH ₃	CH	
SO ₂ C ₂ H ₅	H	H	H	H	OCH ₃	CH ₃	N	
SO ₂ C ₂ H ₅	H	H	H	H	OCH ₃	CH ₃	CH	
SO ₂ C ₂ H ₅	H	H	H	H	CH ₃	CH ₃	N	
SO ₂ C ₂ H ₅	H	7-Br	H	H	CH ₃	CH ₃	CH	
SO ₂ C ₂ H ₅	CH ₃	H	H	H	CH ₃	CH ₃	CH	
SO ₂ C ₂ H ₅	CH ₃	H	H	H	CH ₃	CH ₃	N	
SO ₂ C ₂ H ₅	CH ₃	H	H	H	CH ₃	OCH ₃	N	
SO ₂ C ₂ H ₅	CH ₃	H	H	H	CH ₃	OCH ₃	CH	
SO ₂ C ₂ H ₅	CH ₃	H	H	H	OCH ₃	OCH ₃	N	
SO ₂ C ₂ H ₅	CH ₃	H	H	H	OCH ₃	OCH ₃	CH	
SO ₂ C ₂ H ₅	C ₃ H ₇	H	H	H	OCH ₃	CH ₃	CH	
SO ₂ C ₃ H ₇	H	H	H	H	OCH ₃	OCH ₃	CH	

0 070 698

TABLE I (Continued)

R ₁	R ₂	R ₃	R ₄	R ₅	X	Y	Z	m.p. (°C)
SO ₂ C ₃ H ₇	H	H	H	H	OCH ₃	OCH ₃	N	
SO ₂ C ₃ H ₇	H	H	H	H	OCH ₃	CH ₃	CH	
SO ₂ C ₃ H ₇	H	4-Cl	H	H	OCH ₃	CH ₃	N	
SO ₂ C ₃ H ₇	H	H	H	H	CH ₃	CH ₃	N	
SO ₂ C ₃ H ₇	H	H	H	H	CH ₃	CH ₃	CH	
SO ₂ C ₃ H ₇	CH ₃	H	H	CH ₃	CH ₃	CH ₃	CH	
SO ₂ C ₃ H ₇	CH ₃	H	H	H	CH ₃	CH ₃	N	
SO ₂ C ₃ H ₇	CH ₃	H	H	H	CH ₃	OCH ₃	CH	
SO ₂ C ₃ H ₇	CH ₃	5-OCH ₃	H	H	CH ₃	OCH ₃	N	
SO ₂ C ₃ H ₇	CH ₃	H	H	H	OCH ₃	OCH ₃	CH	
SO ₂ C ₃ H ₇	CH ₃	H	H	H	OCH ₃	OCH ₃	N	
SO ₂ C ₃ H ₇	C ₂ H ₅	H	H	H	OCH ₃	OCH ₃	N	
SO ₂ C ₃ H ₇	i-C ₃ H ₇	4-NO ₂	H	H	CH ₃	OCH ₃	CH	
SO ₂ N(CH ₃) ₂	H	H	H	H	CH ₃	OCH ₃	N	
SO ₂ N(CH ₃) ₂	H	H	H	H	CH ₃	CH ₃	N	
SO ₂ N(CH ₃) ₂	H	H	H	CH ₃	CH ₃	C ₂ H ₅	N	
SO ₂ N(CH ₃) ₂	H	H	H	H	CH ₃	OCH ₃	CH	
SO ₂ N(CH ₃) ₂	H	5-Cl	7-Cl	H	CH ₃	Cl	CH	
SO ₂ N(CH ₃) ₂	CH ₃	H	H	H	OCH ₃	OC ₂ H ₅	CH	
SO ₂ N(CH ₃) ₂	C ₂ H ₅	H	H	H	OCH ₃	OCH ₃	CH	
SO ₂ N(CH ₃) ₂	C ₃ H ₇	H	H	H	OCH ₃	CH ₃	CH	
SO ₂ N(CH ₃) ₂	H	H	H	H	OCH ₃	CH ₃	N	
SO ₂ N(CH ₃)C ₂ H ₅	H	H	H	H	OCH ₃	CH ₃	N	
SO ₂ N(CH ₃)C ₂ H ₅	H	H	H	H	OCH ₃	CH ₃	N	
SO ₂ N(CH ₃)C ₂ H ₅	H	6-Cl	H	H	OCH ₃	N(CH ₃) ₂	N	
SO ₂ N(CH ₃)C ₂ H ₅	H	6-Cl	H	H	CH ₃	OCH ₃	N	
SO ₂ N(CH ₃)C ₂ H ₅	H	6-Cl	H	CH ₃	CH ₃	OCH ₃	N	
SO ₂ N(CH ₃)C ₂ H ₅	H	6-Cl	H	H	CH ₃	OCH ₃	CH	
SO ₂ N(CH ₃)C ₂ H ₅	CH ₃	5-C ₂ H ₅	H	H	CH ₃	OCH ₃	CH	

0 070 698

TABLE I (Continued)

R ₁	R ₂	R ₃	R ₄	R ₅	X	Y	Z	m.p. (°C)
SO ₂ N(CH ₃)C ₂ H ₅	CH ₃	H	H	H	CH ₃	CH ₂ OCH ₃	CH	
SO ₂ N(CH ₃)C ₂ H ₅	CH ₃	H	H	H	CH ₃	CH ₃	CH	
SO ₂ N(CH ₃)C ₂ H ₅	C ₂ H ₅	H	H	H	CH ₃	CH ₃	N	
SO ₂ N(CH ₂ CH ₃) ₂	H	5-NO ₂	H	CH ₃	OCH ₃	CH ₃	N	
SO ₂ N(CH ₂ CH ₃) ₂	H	H	H	H	OCH ₃	OCH ₃	N	
SO ₂ N(CH ₂ CH ₃) ₂	H	H	6-Br	H	OCH ₃	OCH ₃	N	
SO ₂ N(CH ₂ CH ₃) ₂	CH ₃	H	H	H	OCH ₃	Cl	CH	
SO ₂ N(CH ₂ CH ₃) ₂	C ₂ H ₅	H	H	H	CH ₃	CH ₃	CH	
SO ₂ N $\begin{array}{l} \text{CH}_3 \\ \text{CH}(\text{CH}_3)_2 \end{array}$	H	H	H	H	CH ₃	CH ₃	CH	
SO ₂ N $\begin{array}{l} \text{CH}_3 \\ \text{CH}(\text{CH}_3)_2 \end{array}$	H	H	H	H	CH ₃	H	CH	
SO ₂ N $\begin{array}{l} \text{CH}_3 \\ \text{CH}(\text{CH}_3)_2 \end{array}$	H	H	H	H	CH ₃	OCH ₃	N	
SO ₂ N $\begin{array}{l} \text{CH}_3 \\ \text{CH}(\text{CH}_3)_2 \end{array}$	H	H	H	H	CH ₃	N(CH ₃) ₂	N	
SO ₂ N $\begin{array}{l} \text{CH}_3 \\ \text{CH}(\text{CH}_3)_2 \end{array}$	i-C ₃ H ₇	H	5-Br	H	OCH ₃	OCH ₃	N	
SO ₂ N $\begin{array}{l} \text{CH}_3 \\ \text{CH}(\text{CH}_3)_2 \end{array}$	H	7-F	H	H	OCH ₃	OC ₂ H ₅	N	
SO ₂ N $\begin{array}{l} \text{CH}_3 \\ \text{CH}(\text{CH}_3)_2 \end{array}$	H	H	H	H	OCH ₃	CH ₃	N	
CH ₃	SO ₂ C ₆ H ₅	6-F	H	CH ₃	CH ₃	OCH ₃	CH	
CH ₃	SO ₂ C ₆ H ₅	H	H	H	OCH ₃	CH ₃	N	
CH ₃	SO ₂ C ₆ H ₅	H	H	H	CH ₃	CH ₃	CH	
C ₂ H ₅	SO ₂ C ₆ H ₅	H	H	H	CH ₃	N(CH ₃) ₂	N	
C ₂ H ₅	SO ₂ C ₆ H ₅	H	H	H	OCH ₃	CH ₃	N	
i-C ₃ H ₇	SO ₂ C ₆ H ₅	H	H	H	CH ₃	OC ₂ H ₅	CH	

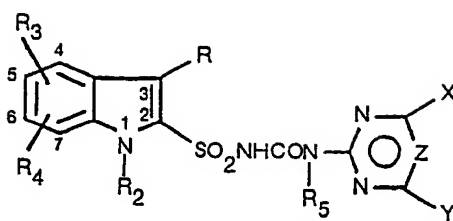
0 070 698

TABLE I (Continued)

R ₁	R ₂	R ₃	R ₄	R ₅	X	Y	Z	m.p. (°C)
CH ₃	SO ₂ C ₆ H ₅	7-NO ₂	H	H	OCH ₃	C ₂ H ₅	N	
CO ₂ CH ₃	H	H	H	H	CH ₃	OCH ₃	CH	
CO ₂ CH ₃	H	H	H	H	CH ₃	CH ₃	CH	
CO ₂ CH ₃	H	H	H	H	OCH ₃	OCH ₃	CH	
CO ₂ CH ₃	H	5-OCH ₃	H	H	OCH ₃	OCH ₃	N	
CO ₂ CH ₃	H	H	H	H	OCH ₃	CH ₃	N	
CO ₂ CH ₃	H	H	H	CH ₃	CH ₃	CH ₃	N	
CO ₂ CH ₃	CH ₃	H	H	CH ₃	CH ₃	CH ₃	N	
CO ₂ CH ₃	CH ₃	6-C ₂ H ₅	H	H	CH ₃	OCH ₃	N	
CO ₂ CH ₃	CH ₃	H	H	H	OCH ₃	OCH ₃	N	
CO ₂ CH ₃	CH ₃	H	H	H	OCH ₃	OCH ₃	CH	
CO ₂ CH ₃	CH ₃	H	H	H	OCH ₃	CH ₃	CH	200-203°(d)
CO ₂ CH ₃	H	H	H	H	OCH ₃	Cl	CH	
CO ₂ CH ₃	H	H	H	H	OCH ₃	N(CH ₃) ₂	CH	
CO ₂ CH ₃	H	H	H	H	OCH ₃	C ₂ H ₅	CH	
CO ₂ CH ₃	H	H	H	H	OCH ₃	OC ₂ H ₅	N	

0 070 698

TABLE II



R	R ₂	R ₃	R ₄	R ₅	X	Y	Z	m.p. (°C)
H	H	4-F	H	CH ₃	CH ₃	CH ₃	N	
H	H	H	H	H	CH ₃	CH ₃	CH	
H	H	H	H	H	CH ₃	OCH ₃	N	
H	H	H	H	H	CH ₃	OCH ₃	CH	
H	H	H	H	H	OCH ₃	OCH ₃	CH	
H	H	H	H	H	OCH ₃	OCH ₃	N	
H	CH ₃	H	H	H	OCH ₃	OCH ₃	CH	
H	CH ₃	5-Cl	H	H	OCH ₃	OCH ₃	N	
H	CH ₃	H	H	H	OCH ₃	CH ₃	N	
H	CH ₃	H	H	H	OCH ₃	CH ₃	CH	
H	CH ₃	H	4-Br	H	CH ₃	CH ₃	N	
H	CH ₃	H	H	H	CH ₃	CH ₃	CH	
H	C ₂ H ₅	H	H	CH ₃	CH ₃	CH ₃	N	
H	i-C ₃ H ₇	H	H	H	CH ₃	OC ₂ H ₅	CH	
H	SO ₂ C ₆ H ₅	H	H	H	CH ₃	CH ₃	CH	
H	SO ₂ C ₆ H ₅	H	H	H	CH ₃	OCH ₃	N	
H	SO ₂ C ₆ H ₅	H	H	H	CH ₃	OCH ₃	CH	
H	SO ₂ C ₆ H ₅	H	H	H	OCH ₃	OCH ₃	N	
H	SO ₂ C ₆ H ₅	H	H	H	OCH ₃	OCH ₃	CH	
H	SO ₂ C ₆ H ₅	6-NO ₂	H	H	OCH ₃	Cl	CH	
H	SO ₂ C ₆ H ₅	H	H	H	CH ₃	N(CH ₃) ₂	CH	
CH ₃	H	H	5-Br	H	OCH ₃	OCH ₃	CH	

0 070 698

TABLE II (Continued)

R	R ₂	R ₃	R ₄	R ₅	X	Y	Z	m.p. (°C)
CH ₃	H	H	H	H	OCH ₃	OCH ₃	N	
CH ₃	H	6-CH ₃	H	H	OCH ₃	CH ₂ OCH ₃	N	
CH ₃	H	H	H	H	OCH ₃	CH ₃	N	
CH ₃	H	H	H	H	OCH ₃	CH ₃	CH	
CH ₃	H	H	H	H	CH ₃	CH ₃	N	
CH ₃	H	6-C ₃ H ₇	H	H	CH ₃	CH ₃	CH	
CH ₃	CH ₃	H	H	H	CH ₃	CH ₃	CH	
CH ₃	CH ₃	H	H	CH ₃	CH ₃	CH ₃	N	
CH ₃	CH ₃	H	H	H	OCH ₃	CH ₃	N	
CH ₃	CH ₃	H	H	H	OCH ₃	CH ₃	CH	
CH ₃	CH ₃	5-OC ₃ H ₇	H	H	OCH ₃	OCH ₃	N	
CH ₃	CH ₃	H	H	H	OCH ₃	OCH ₃	CH	
CH ₃	C ₂ H ₅	H	H	CH ₃	OCH ₃	CH ₃	CH	
CH ₃	n-C ₃ H ₇	H	H	H	OCH ₃	CH ₃	N	
CH ₃	C ₂ H ₅	4-Br	H	H	CH ₃	H	CH	
CH ₃	CH ₃	4-Cl	6-Cl	H	CH ₃	Cl	CH	
CH ₃	SO ₂ C ₆ H ₅	H	H	H	CH ₃	OCH ₃	CH	
CH ₃	SO ₂ C ₆ H ₅	H	H	H	CH ₃	OCH ₃	N	
CH ₃	SO ₂ C ₆ H ₅	H	H	H	OCH ₃	OCH ₃	CH	
CH ₃	SO ₂ C ₆ H ₅	H	H	H	OCH ₃	OCH ₃	N	
CH ₃	SO ₂ C ₆ H ₅	H	H	H	CH ₃	CH ₃	N	
CH ₃	SO ₂ C ₆ H ₅	H	H	CH ₃	CH ₃	C ₂ H ₅	N	
CH ₃	SO ₂ C ₆ H ₅	5-NO ₂	H	H	CH ₃	OCH ₃	N	
C ₂ H ₅	H	H	H	H	CH ₃	OCH ₃	N	
C ₂ H ₅	H	H	H	H	CH ₃	OCH ₃	CH	
C ₂ H ₅	H	H	H	H	CH ₃	OCH ₃	CH	
C ₂ H ₅	C ₂ H ₅	4-OCH ₃	H	H	CH ₃	OC ₂ H ₅	CH	
C ₂ H ₅	C ₂ H ₅	H	H	CH ₃	CH ₃	OCH ₃	CH	
C ₂ H ₅	i-C ₃ H ₇	H	H	H	CH ₃	N(CH ₃) ₂	N	

0 070 698

TABLE II (Continued)

R	R ₂	R ₃	R ₄	R ₅	X	Y	Z	m.p. (°C)
C ₂ H ₅	SO ₂ C ₆ H ₅	H	H	H	OCH ₃	OCH ₃	N	
C ₂ H ₅	SO ₂ C ₆ H ₅	H	H	H	OCH ₃	OCH ₃	N	
C ₂ H ₅	SO ₂ C ₆ H ₅	H	H	H	OCH ₃	CH ₃	N	
C ₂ H ₅	SO ₂ C ₆ H ₅	H	5-Cl	H	OCH ₃	CH ₃	N	
n-C ₃ H ₇	H	H	H	H	OCH ₃	CH ₃	N	
n-C ₃ H ₇	H	H	H	H	OCH ₃	CH ₃	CH	
n-C ₃ H ₇	CH ₃	H	H	H	OCH ₃	CH ₂ OCH ₃	CH	
n-C ₃ H ₇	C ₃ H ₇	6-F	H	H	OCH ₃	OCH ₃	CH	
n-C ₃ H ₇	SO ₂ C ₆ H ₅	H	H	CH ₃	OCH ₃	OCH ₃	CH	
n-C ₃ H ₇	SO ₂ C ₆ H ₅	H	H	H	CH ₃	N(CH ₃) ₂	CH	
i-C ₃ H ₇	H	H	H	CH ₃	OCH ₃	C ₂ H ₅	N	
i-C ₃ H ₇	H	H	H	H	OCH ₃	CH ₃	N	
i-C ₃ H ₇	H	H	H	H	OCH ₃	CH ₃	N	
i-C ₃ H ₇	CH ₃	H	H	H	OCH ₃	CH ₃	N	
n-C ₃ H ₇	H	H	H	H	CH ₃	CH ₃	CH	
n-C ₃ H ₇	H	H	H	H	CH ₃	N(CH ₃) ₂	CH	
n-C ₃ H ₇	H	5-Br	7-Br	H	CH ₃	CH ₃	CH	
n-C ₃ H ₇	i-C ₃ H ₇	H	H	H	CH ₃	CH ₃	CH	
n-C ₄ H ₉	H	H	H	H	CH ₃	OC ₂ H ₅	N	
n-C ₄ H ₉	H	H	H	H	OCH ₃	OCH ₃	N	
n-C ₄ H ₉	C ₂ H ₅	H	H	H	OCH ₃	OCH ₃	N	
s-C ₄ H ₉	H	H	H	H	OCH ₃	Cl	CH	
s-C ₄ H ₉	H	6-OC ₂ H ₅	H	CH ₃	OCH ₃	CH ₃	CH	
s-C ₄ H ₉	H	H	H	H	OCH ₃	CH ₃	CH	
t-C ₄ H ₉	H	H	H	H	OCH ₃	H	CH	
t-C ₄ H ₉	CH ₃	H	4-Cl	H	OCH ₃	CH ₃	N	
t-C ₄ H ₉	SO ₂ C ₆ H ₅	H	H	H	CH ₃	OCH ₃	N	
i-C ₄ H ₉	SO ₂ C ₆ H ₅	H	H	H	CH ₃	CH ₂ OCH ₃	N	
n-C ₄ H ₉	SO ₂ C ₆ H ₅	H	H	H	CH ₃	CH ₃	N	

0 070 698

TABLE II (Continued)

R ₁	R ₂	R ₃	R ₄	R ₅	X	Y	Z	m.p. (°C)
s-C ₄ H ₉	SO ₂ C ₆ H ₅	H	H	H	OCH ₃	CH ₃	CH	
t-C ₄ H ₉	SO ₂ C ₆ H ₅	H	H	H	OCH ₃	OCH ₃	CH	
i-C ₄ H ₉	SO ₂ C ₆ H ₅	H	H	H	OCH ₃	OCH ₃	CH	
CH ₂ CO ₂ CH ₃	H	H	H	H	OCH ₃	OCH ₃	CH	
CH ₂ CO ₂ CH ₃	H	H	H	CH ₃	OCH ₃	OCH ₃	N	
CH ₂ CO ₂ CH ₃	H	H	H	H	OCH ₃	CH ₃	CH	
CH ₂ CO ₂ CH ₃	H	H	H	H	OCH ₃	CH ₃	N	
CH ₂ CO ₂ CH ₃	H	H	H	H	CH ₃	CH ₃	N	
CH ₂ CO ₂ CH ₃	H	6-OCH ₃	H	H	CH ₃	CH ₃	CH	
CH ₂ CO ₂ CH ₃	H	H	H	H	CH ₃	CH ₃	CH	
CH ₂ CO ₂ CH ₃	CH ₃	H	H	H	CH ₃	CH ₃	N	
CH ₂ CO ₂ CH ₃	CH ₃	H	H	H	CH ₃	CH ₃	CH	
CH ₂ CO ₂ CH ₃	CH ₃	H	H	H	CH ₃	CH ₂ OCH ₃	N	
CH ₂ CO ₂ CH ₃	CH ₃	H	H	H	OCH ₃	OCH ₃	N	
CH ₂ CO ₂ CH ₃	CH ₃	H	H	H	OCH ₃	OCH ₃	CH	
CH ₂ CO ₂ CH ₃	CH ₃	H	H	H	OCH ₃	CH ₃	N	
CH ₂ CO ₂ CH ₃	CH ₃	5-Me	H	H	OCH ₃	CH ₃	CH	
CH ₂ CO ₂ CH ₃	C ₂ H ₅	H	7-Cl	CH ₃	OCH ₃	OC ₂ H ₅	CH	
CH ₂ CO ₂ CH ₃	C ₂ H ₅	H	H	H	CH ₃	Cl	CH	
CH ₂ CO ₂ CH ₃	i-C ₃ H ₇	H	H	H	CH ₃	OCH ₃	CH	
CH ₂ CO ₂ CH ₃	n-C ₃ H ₇	7-F	H	H	CH ₃	OCH ₃	N	
CH ₂ CO ₂ C ₂ H ₅	H	H	H	H	CH ₃	CH ₃	CH	
CH ₂ CO ₂ C ₂ H ₅	H	H	H	H	OCH ₃	CH ₃	CH	
CH ₂ CO ₂ C ₂ H ₅	H	H	H	H	OCH ₃	CH ₃	N	
CH ₂ CO ₂ C ₂ H ₅	C ₂ H ₅	H	6-Br	CH ₃	OCH ₃	CH ₃	N	
CH ₂ CO ₂ C ₂ H ₅	C ₂ H ₅	H	H	H	OCH ₃	CH ₃	N	
CH ₂ CO ₂ i-C ₃ H ₇	H	H	H	H	OCH ₃	CH ₃	N	
CH ₂ CO ₂ i-C ₃ H ₇	H	H	H	H	CH ₃	C ₂ H ₅	CH	
CH ₂ CO ₂ i-C ₃ H ₇	H	4-NO ₂	H	H	CH ₃	N(CH ₃) ₂	CH	

0 070 698

TABLE II (Continued)

R	R ₂	R ₃	R ₄	R ₅	X	Y	Z	m.p. (°C)
CH ₂ CO ₂ -i-C ₃ H ₇	CH ₃	H	H	H	CH ₃	H	CH	
CH ₂ CO ₂ n-C ₃ H ₇	H	H	H	H	CH ₃	OCH ₃	CH	
CH ₂ CO ₂ n-C ₃ H ₇	H	H	H	CH ₃	OCH ₃	OCH ₃	N	
CH ₂ CO ₂ H	H	H	H	CH ₃	OCH ₃	OCH ₃	N	
CH ₂ CO ₂ H	H	H	H	H	OCH ₃	OCH ₃	CH	
CH ₂ CO ₂ H	H	H	H	H	OCH ₃	CH ₃	CH	
CH ₂ CO ₂ H	H	6-OC ₂ H ₅	H	H	OCH ₃	CH ₃	N	
CH ₂ CO ₂ H	H	H	H	H	CH ₃	CH ₃	CH	
CH ₂ CO ₂ H	H	H	H	H	OCH ₃	CH ₃	N	
CH ₂ CO ₂ H	CH ₃	H	H	H	CH ₃	OCH ₃	CH	
CH ₂ CO ₂ H	CH ₃	H	H	H	OCH ₃	OCH ₃	N	
CH ₂ CO ₂ H	CH ₃	H	H	H	OCH ₃	OCH ₃	CH	
CH ₂ CO ₂ H	C ₂ H ₅	7-NO ₂	H	CH ₃	OCH ₃	OCH ₃	CH	
CO ₂ CH ₃	H	H	H	H	OCH ₃	CH ₃	N	
CO ₂ CH ₃	H	H	H	H	OCH ₃	CH ₃	CH	
CO ₂ CH ₃	H	H	H	H	OCH ₃	OCH ₃	CH	
CO ₂ CH ₃	H	H	H	H	OCH ₃	OCH ₃	N	
CO ₂ CH ₃	H	H	H	H	CH ₃	CH ₃	CH	
CO ₂ CH ₃	H	7-Br	6-Br	H	CH ₃	Cl	CH	
CO ₂ CH ₃	CH ₃	H	H	CH ₃	CH ₃	C ₂ H ₅	CH	
CO ₂ CH ₃	CH ₃	H	H	H	CH ₃	OCH ₃	CH	
CO ₂ CH ₃	C ₂ H ₅	H	H	H	CH ₃	OCH ₃	CH	
CO ₂ CH ₃	i-C ₃ H ₇	H	H	H	OCH ₃	H	CH	
CO ₂ C ₂ H ₅	H	H	H	H	OCH ₃	OCH ₃	N	
CO ₂ C ₂ H ₅	H	H	H	H	OCH ₃	OCH ₃	N	
CO ₂ C ₃ H ₅	H	H	H	H	OCH ₃	OCH ₃	N	
CO ₂ C ₂ H ₅	H	H	H	CH ₃	OCH ₃	OCH ₃	N	
CO ₂ C ₂ H ₅	H	4-C ₂ H ₅	H	H	CH ₃	CH ₂ OCH ₃	CH	
CO ₂ C ₂ H ₅	CH ₃	H	H	H	CH ₃	OCH ₃	CH	

0 070 698

TABLE II (Continued)

R	R ₂	R ₃	R ₄	R ₅	X	Y	Z	m.p. (°C)
CO ₂ C ₂ H ₅	CH ₃	H	H	H	CH ₃	OCH ₃	CH	
CO ₂ C ₂ H ₅	n-C ₃ H ₇	H	H	H	OCH ₃	OCH ₃	CH	
CO ₂ i-C ₃ H ₇	H	H	H	H	OCH ₃	C ₂ H ₅	N	
CO ₂ i-C ₃ H ₇	H	H	H	H	OCH ₃	CH ₃	N	
CO ₂ i-C ₃ H ₇	H	H	5-Cl	H	OCH ₃	OC ₂ H ₅	N	
CO ₂ i-C ₃ H ₇	H	7-OCH ₃	H	H	OCH ₃	Cl	CH	
CO ₂ i-C ₃ H ₇	CH ₃	H	H	H	CH ₃	H	CH	
CO ₂ i-C ₃ H ₇	CH ₃	H	H	H	CH ₃	CH ₃	CH	
CO ₂ n-C ₃ H ₇	H	H	H	CH ₃	CH ₃	CH ₃	CH	
CO ₂ n-C ₃ H ₇	H	H	H	H	CH ₃	CH ₃	N	
CO ₂ n-C ₃ H ₇	H	H	H	H	CH ₃	N(CH ₃) ₂	N	
CO ₂ n-C ₃ H ₇	H	H	H	H	CH ₃	OCH ₃	N	
CO ₂ n-C ₃ H ₇	C ₂ H ₅	6-Cl	H	H	CH ₃	OCH ₃	N	
CO ₂ n-C ₃ H ₇	n-C ₃ H ₇	H	H	H	CH ₃	OCH ₃	N	
CO ₂ H	H	H	H	H	OCH ₃	CH ₃	N	
CO ₂ H	H	H	H	H	OCH ₃	CH ₃	CH	
CO ₂ H	H	H	H	H	OCH ₃	OCH ₃	N	
CO ₂ H	H	H	H	H	OCH ₃	OCH ₃	CH	
CO ₂ H	H	H	H	CH ₃	CH ₃	Cl	CH	
CO ₂ H	C ₂ H ₅	H	H	H	CH ₃	C ₂ H ₅	CH	
CO ₂ H	i-C ₃ H ₇	6-Cl	H	H	CH ₃	OCH ₃	CH	
CO ₂ H	n-C ₃ H ₇	H	5-Br	H	CH ₃	H	CH	
(CH ₂) ₂ CO ₂ CH ₃	H	H	H	H	OCH ₃	OCH ₃	N	
(CH ₂) ₂ CO ₂ CH ₃	H	H	H	H	OCH ₃	OCH ₃	CH	
(CH ₂) ₂ CO ₂ CH ₃	H	H	H	H	OCH ₃	CH ₃	CH	
(CH ₂) ₂ CO ₂ CH ₃	H	H	H	H	OCH ₃	CH ₃	N	
(CH ₂) ₂ CO ₂ CH ₃	H	H	H	H	OCH ₃	OC ₂ H ₅	N	
(CH ₂) ₂ CO ₂ CH ₃	H	7-NO ₂	H	H	CH ₃	CH ₃	CH	
(CH ₂) ₂ CO ₂ CH ₃	H	H	6-Cl	CH ₃	CH ₃	CH ₃	N	

TABLE II (Continued)

R	R ₂	R ₃	R ₄	R ₅	X	Y	Z	m.p. (°C)
(CH ₂) ₂ CO ₂ CH ₃	CH ₃	H	H	H	CH ₃	CH ₃	N	
(CH ₂) ₂ CO ₂ CH ₃	CH ₃	H	H	H	CH ₃	CH ₃	N	
(CH ₂) ₂ CO ₂ CH ₃	C ₂ H ₅	H	H	H	CH ₃	CH ₂ OCH ₃	N	
(CH ₂) ₂ CO ₂ CH	H	H	H	H	OCH ₃	Cl	CH	
(CH ₂) ₂ CO ₂ CH ₃	H	H	H	H	OCH ₃	N(CH ₃) ₂	CH	
(CH ₂) ₂ CO ₂ CH ₃	H	H	H	H	OCH ₃	OCH ₃	CH	
(CH ₂) ₂ CO ₂ CH ₃	H	H	H	H	OCH ₃	OCH ₃	CH	
(CH ₂) ₂ CO ₂ CH ₃	H	H	H	CH ₃	OCH ₃	OCH ₃	N	
(CH ₂) ₂ CO ₂ CH ₃	C ₂ H ₅	7-Cl	H	H	CH ₃	OCH ₃	N	
(CH ₂) ₂ CO ₂ CH ₃	C ₃ H ₇	H	H	H	CH ₃	OCH ₃	N	
(CH ₂) ₂ CO ₂ i-C ₃ H ₇	H	H	H	H	CH ₃	C ₂ H ₅	N	
(CH ₂) ₂ CO ₂ i-C ₃ H ₇	H	H	H	H	CH ₃	C ₂ H ₅	CH	
(CH ₂) ₂ CO ₂ i-C ₃ H ₇	H	H	H	H	OCH ₃	CH ₃	CH	
(CH ₂) ₂ CO ₂ i-C ₃ H ₇	H	H	H	H	OCH ₃	CH ₃	CH	
(CH ₂) ₂ CO ₂ n-C ₃ H ₇	H	H	H	CH ₃	OCH ₃	OC ₂ H ₅	CH	
(CH ₂) ₂ CO ₂ n-C ₃ H ₇	H	5-F	H	H	OCH ₃	OC ₂ H ₅	N	
(CH ₂) ₂ CO ₂ n-C ₃ H ₇	i-C ₃ H ₇	H	6-Br	H	OCH ₃	N(CH ₃) ₂	N	
(CH ₂) ₂ CO ₂ n-C ₃ H ₇	CH ₃	H	H	H	OCH ₃	CH ₃	N	
(CH ₂) ₂ CO ₂ H	H	H	H	H	OCH ₃	OCH ₃	N	
(CH ₂) ₂ CO ₂ H	H	H	H	H	OCH ₃	OCH ₃	CH	
(CH ₂) ₂ CO ₂ H	H	H	H	H	OCH ₃	CH ₃	CH	
(CH ₂) ₂ CO ₂ H	H	H	H	H	OCH ₃	CH ₃	N	
(CH ₂) ₂ CO ₂ H	H	6-Br	H	H	OCH ₃	CH ₃	N	
(CH ₂) ₂ CO ₂ H	CH ₃	H	H	H	CH ₃	CH ₃	N	
(CH ₂) ₂ CO ₂ H	n-C ₃ H ₇	H	H	CH ₃	CH ₃	CH ₃	N	
CH ₂ OC ₂ H ₅	H	H	H	H	CH ₃	CH ₃	CH	
CH ₂ OC ₂ H ₅	H	H	H	H	CH ₃	OCH ₃	CH	
CH ₂ OC ₂ H ₅	H	H	H	H	CH ₃	OCH ₃	N	
CH ₂ OC ₂ H ₅	H	H	H	H	OCH ₃	OCH ₃	N	

0 070 698

TABLE II (Continued)

R	R ₂	R ₃	R ₄	R ₅	X	Y	Z	m.p. (°C)
CH ₂ OC ₂ H ₅	H	H	H	H	OCH ₃	N(CH ₃) ₂	N	
CH ₂ OC ₂ H ₅	H	H	5-Br	H	OCH ₃	C ₂ H ₅	N	
CH ₂ OC ₂ H ₅	CH ₃	H	H	H	OCH ₃	H	N	
CH ₂ OC ₂ H ₅	CH ₃	H	H	H	OCH ₃	CH ₂ OCH ₃	N	
CH ₂ OC ₂ H ₅	i-C ₃ H ₇	7-i-C ₃ H ₇	H	H	CH ₃	Cl	CH	
CH ₂ OCH ₃	H	H	H	CH ₃	CH ₃	OCH ₃	CH	
CH ₂ OCH ₃	H	H	H	H	CH ₃	OCH ₃	N	
CH ₂ OCH ₃	H	H	H	H	CH ₃	CH ₃	N	
CH ₂ OCH ₃	H	H	H	H	CH ₃	CH ₃	CH	
CH ₂ OCH ₃	H	H	H	H	OCH ₃	OCH ₃	CH	
CH ₂ OCH ₃	H	H	H	H	OCH ₃	OCH ₃	N	
CH ₂ OCH ₃	H	H	H	H	OCH ₃	OCH ₃	N	
CH ₂ OCH ₃	C ₂ H ₅	4-O-i-C ₃ H ₇	H	H	OCH ₃	OC ₂ H ₅	N	
CH ₂ OCH ₃	C ₂ H ₅	H	H	H	OCH ₃	OCH ₃	N	
CHO	H	H	H	H	CH ₃	OCH ₃	N	
CHO	H	H	H	H	CH ₃	OCH ₃	CH	
CHO	H	H	H	H	CH ₃	CH ₃	CH	
CHO	H	H	H	CH ₃	CH ₃	OCH ₃	CH	
CHO	H	H	H	CH ₃	CH ₃	CH ₂ OCH ₃	CH	
CHO	CH ₃	H	7-Cl	H	OCH ₃	Cl	CH	
CHO	CH ₃	H	H	H	OCH ₃	OCH ₃	CH	
CHO	CH ₃	5-OCH ₃	H	H	OCH ₃	N(CH ₃) ₂	CH	
SO ₂ CH ₃	H	H	H	H	OCH ₃	CH ₃	N	
SO ₂ CH ₃	H	H	H	H	OCH ₃	CH ₃	CH	
SO ₂ CH ₃	H	H	H	H	OCH ₃	OCH ₃	CH	
SO ₂ CH ₃	H	H	H	H	OCH ₃	OCH ₃	N	
SO ₂ CH ₃	H	5-C ₂ H ₅	H	H	CH ₃	OC ₂ H ₅	N	
SO ₂ CH ₃	H	H	H	CH ₃	CH ₃	Cl	CH	
SO ₂ CH ₃	CH ₃	H	H	H	CH ₃	C ₂ H ₅	CH	

0 070 698

TABLE II (Continued)

R	R ₂	R ₃	R ₄	R ₅	X	Y	Z	m.p. (°C)
SO ₂ CH ₃	i-C ₃ H ₇	H	7-Br	H	CH ₃	H	CH	
SO ₂ C ₂ H ₅	i-C ₃ H ₇	H	H	H	OCH ₃	CH ₃	CH	
SO ₂ C ₂ H ₅	i-C ₃ H ₇	H	H	H	OCH ₃	CH	N	
SO ₂ C ₂ H ₅	i-C ₃ H ₇	H	H	H	OCH ₃	CH ₃	N	
SO ₂ C ₂ H ₅	C ₂ H ₅	H	H	H	OCH ₃	CH ₂ OCH ₃	N	
SO ₂ C ₂ H ₅	C ₂ H ₅	H	H	H	OCH ₃	OCH ₃	N	
SO ₂ i-C ₃ H ₇	H	H	H	CH ₃	CH ₃	N(CH ₃) ₂	N	
SO ₂ i-C ₃ H ₇	H	H	H	H	CH ₃	OCH ₃	N	
SO ₂ i-C ₃ H ₇	H	H	H	H	CH ₃	OCH ₃	CH	
SO ₂ i-C ₃ H ₇	n-C ₃ H ₇	6-OCH ₃	H	H	CH ₃	OCH ₃	CH	
SO ₂ -n-C ₃ H ₇	H	H	H	H	CH ₃	OCH ₃	CH	
SO ₂ -n-C ₃ H ₇	H	H	H	H	CH ₃	C ₂ H ₅	N	
SO ₂ -n-C ₃ H ₇	H	H	H	H	OCH ₃	CH ₃	N	
SO ₂ -n-C ₃ H ₇	CH ₃	H	H	H	OCH ₃	CH ₃	N	
SO ₂ N(CH ₃) ₂	H	H	H	H	OCH ₃	CH ₃	N	
SO ₂ N(CH ₃) ₂	H	H	H	H	OCH ₃	CH ₃	N	
SO ₂ N(CH ₃) ₂	H	H	H	H	OCH ₃	CH ₂ OCH ₃	CH	
SO ₂ N(CH ₃) ₂	H	H	H	H	OCH ₃	OCH ₃	CH	
SO ₂ N(CH ₃) ₂	H	H	H	H	CH ₃	OCH ₃	CH	
SO ₂ N(CH ₃) ₂	CH ₃	H	H	CH ₃	CH ₃	OCH ₃	CH	
SO ₂ N(CH ₃) ₂	CH ₃	H	H	H	CH ₃	OCH ₃	N	
SO ₂ N(CH ₃) ₂	i-C ₃ H ₇	4-CH ₃	H	H	CH ₃	OCH ₃	N	
SO ₂ N(CH ₃) ₂	C ₂ H ₅	6-Cl	4-Cl	H	CH ₃	N(CH ₃) ₂	N	
SO ₂ N(CH ₃)C ₂ H ₅	H	H	H	H	OCH ₃	C ₂ H ₅	N	
SO ₂ N(CH ₃)C ₂ H ₅	H	H	H	H	OCH ₃	OCH ₃	N	
SO ₂ N(CH ₃)C ₂ H ₅	H	H	H	H	CH ₃	OCH ₃	N	
SO ₂ N(CH ₃)C ₂ H ₅	H	H	H	H	CH ₃	OCH ₃	N	
SO ₂ N(CH ₃)C ₂ H ₅	H	H	H	H	CH ₃	OCH ₃	CH	
SO ₂ N(CH ₃)C ₂ H ₅	C ₂ H ₅	7-OiC ₃ H ₇	H	H	CH ₃	OCH ₃	CH	

0 070 698

TABLE II (Continued)

R	R ₂	R ₃	R ₄	R ₅	X	Y	Z	m.p. (°C)
SO ₂ N(CH ₃)C ₂ H ₅	n-C ₃ H ₇	H	H	H	CH ₃	N(CH ₃) ₂	CH	
SO ₂ N(C ₂ H ₅) ₂	H	H	H	H	OCH ₃	CH ₃	CH	
SO ₂ N(C ₂ H ₅) ₂	H	H	H	CH ₃	OCH ₃	CH ₃	CH	
SO ₂ N(C ₂ H ₅) ₂	H	H	H	H	OCH ₃	CH ₃	N	
SO ₂ N(C ₂ H ₅) ₂	H	H	H	H	OCH ₃	H	N	
SO ₂ N(C ₂ H ₅) ₂	C ₂ H ₅	H	H	H	OCH ₃	C ₂ H ₅	N	
SO ₂ N(C ₂ H ₅) ₂	CH ₃	H	5-Br	H	CH ₃	OC ₂ H ₅	N	
SO ₂ N(C ₂ H ₅) ₂	CH ₃	4-CH ₃	H	H	CH ₃	OCH ₃	N	
SO ₂ N(CH ₃)C ₃ H ₇	H	H	H	H	CH ₃	CH ₂ OCH ₃	N	
SO ₂ N(CH ₃)C ₃ H ₇	H	H	H	H	CH ₃	OCH ₃	N	
SO ₂ N(CH ₃)C ₃ H ₇	H	H	H	CH ₃	OCH ₃	OCH ₃	N	
SO ₂ N(CH ₃)C ₃ H ₇	H	H	H	H	OCH ₃	CH ₃	CH	
SO ₂ N(CH ₃)C ₃ H ₇	n-C ₃ H ₇	H	H	H	OCH ₃	Cl	CH	
SO ₂ N(CH ₃) <i>i</i> -C ₃ H ₇	n-C ₃ H ₇	H	H	H	OCH ₃	CH ₃	CH	
SO ₂ N(CH ₃) <i>i</i> -C ₃ H ₇	n-C ₃ H ₇	H	7-Cl	H	CH ₃	CH ₃	CH	
SO ₂ N(CH ₃) <i>i</i> -C ₃ H ₇	n-C ₃ H ₇	H	H	H	CH ₃	OCH ₃	N	
SO ₂ N(CH ₃) <i>i</i> -C ₃ H ₇	C ₂ H ₅	5-NO ₂	H	H	CH ₃	OCH ₃	N	
CH ₂ N(CH ₃) ₂	H	H	H	H	CH ₃	OCH ₃	N	
CH ₂ N(CH ₃) ₂	H	H	H	H	CH ₃	OCH ₃	CH	
CH ₂ N(CH ₃) ₂	H	H	H	H	OCH ₃	OCH ₃	CH	
CH ₂ N(CH ₃) ₂	H	H	H	H	OCH ₃	OCH ₃	N	
CH ₂ N(CH ₃) ₂	H	H	H	H	OCH ₃	N(CH ₃) ₂	N	
CH ₂ N(CH ₃) ₂	H	H	H	CH ₃	OCH ₃	CH ₃	N	
CH ₂ N(CH ₃) ₂	CH ₃	H	H	CH ₃	OCH ₃	CH ₃	N	
CH ₂ N(CH ₃) ₂	C ₂ H ₅	H	H	H	OCH ₃	Cl	CH	
CH ₂ N(CH ₃) ₂	<i>i</i> -C ₃ H ₇	H	H	H	OCH ₃	OC ₂ H ₅	CH	

Formulations

Useful formulations of the compounds of Formula I can be prepared in conventional ways. They include dusts, granules, pellets, solutions, suspensions, emulsions, wettable powders, emulsifiable concentrates and the like. Many of these may be applied directly. Sprayable formulations can be extended in suitable media and used at spray volumes of from a few liters to several hundred liters per hectare. High strength compositions are primarily used as intermediates for further formulation. The formulations, broadly, contain about 0.1% to 99% by weight of active ingredient(s) and at least one of (a) about 0.1% to 20% surfactant(s) and (b) about 1% to 99.9% solid or liquid diluent(s). More specifically, they will contain these ingredients in the following approximate proportions:

TABLE 3

	Active Ingredient	Weight Percent*	
		Diluent(s)	Surfactant(s)
Wettable Powders	20—90	0—74	1—10
Oil Suspensions, Emulsions, Solutions, (including Emulsifiable Concentrates	3—50	40—95	0—15
Aqueous Suspension	10—50	40—84	1—20
Dusts	1—25	70—99	0—5
Granules and Pellets	0.1—95	5—99.9	0—15
High Strength Compositions	90—99	0—10	0—2

*Active ingredient plus at least one of a Surfactant or a Diluent equals 100 weight percent.

Lower or higher levels of active ingredient can, of course, be present depending on the intended use and the physical properties of the compound. Higher ratios of surfactant to active ingredient are sometimes desirable, and are achieved by incorporation into the formulation or by tank mixing.

Typical solid diluents are described in Watkins, et al., "Handbook of Insecticide Dust Diluents and Carriers", 2nd Ed., Dorland Books, Caldwell, New Jersey, but other solids, either mined or manufactured, may be used. The more absorptive diluents are preferred for wettable powders and the denser ones for dusts. Typical liquid diluents and solvents are described in Marsden, "Solvents Guide", 2nd Ed., Interscience, New York, 1950. Solubility under 0.1% is preferred for suspension concentrates; solution concentrates are preferably stable against phase separation at 0°C. "McCutcheon's Detergents and Emulsifiers Annual", MC Publishing Corp., Ridgewood, New Jersey, as well as Sisely and Wood, "Encyclopedia of Surface Active Agents", Chemical Publishing Co., Inc., New York, 1964, list surfactants and recommended uses. All formulations can contain minor amounts of additives to reduce foaming, caking, corrosion, microbiological growth, etc.

The methods of making such compositions are well known. Solutions are prepared by simply mixing the ingredients. Fine solid compositions are made by blending and, usually, grinding as in a hammer or fluid energy mill. Suspensions are prepared by wet milling (see, for example, Littler, U.S. Patent 3,060,084). Granules and pellets may be made by spraying the active material upon preformed granular carriers or by agglomeration techniques. See J. E. Browning, "Agglomeration", *Chemical Engineering*, December 4, 1967, pp. 147ff. and "Perry's Chemical Engineer's Handbook", 5th Ed., McGraw-Hill, New York, 1973, pp. 8—57ff.

For further information regarding the art of formulation, see for example:

H. M. Loux, U.S. Patent 3,235,361, February 15, 1966, Col. 6, line 16 through Col. 7, line 19 and Examples 10 through 41;

R. W. Luckenbaugh, U.S. Patent 3,309,192, March 14, 1967, Col. 5, line 43 through Col. 7, line 62 and Examples 8, 12, 15, 39, 41, 52, 53, 58, 132, 138—140, 162—164, 166, 167 and 169—182;

H. Gysin and E. Knusli, U.S. Patent 2,891,855, June 23, 1959, Col. 3, line 66 through Col. 5, line 17 and Examples 1—4;

G. C. Klingman, "Weed Control as a Science", John Wiley & Sons, Inc., New York, 1961, pp. 81—96; and

J. D. Fryer and S. A. Evans, "Weed Control Handbook", 5th Ed., Blackwell Scientific Publications, Oxford, 1968, pp. 101—103.

In the following examples, all parts are by weight unless otherwise indicated.

0 070 698

Example 8

Wettable Powder

5	3-[[[4-methoxy-6-methylpyrimidin-2-yl]aminocarbonyl]aminosulfonyl]-1-methyl-1H-indole-2-carboxylic acid, methyl ester	80%
	sodium alkyl naphthalenesulfonate	2%
	sodium ligninsulfonate	2%
10	synthetic amorphous silica	3%
	kaolinite	13%

15 The ingredients are blended, hammer-milled until all the solids are essentially under 50 microns reblended, and packaged.

Example 9

Wettable Powder

20	3-[[[4,6-dimethyl-1,3,5-triazin-2-yl]-aminocarbonyl]aminosulfonyl]-1-methyl-1H-indole-2-carboxylic acid, methyl ester	50%
	sodium alkyl naphthalenesulfonate	2%
25	low viscosity methyl cellulose	2%
	diatomaceous earth	46%

30 The ingredients are blended, coarsely hammer-milled and then air-milled to produce particles essentially all below 10 microns in diameter. The product is reblended before packaging.

Example 10

Granule

35	Wettable Powder of Example 9	5%
	attapulgitic granules	95%
40	(U.S.S. 20—40 mesh; 0.84—0.42 mm)	

A slurry of wettable powder containing 25% solids is sprayed on the surface of attapulgitic granules in a double-cone blender. The granules are dried and packaged.

Example 11

Extruded Pellet

45	3-[[[4,6-dimethoxy-1,3,5-triazin-2-yl]aminocarbonyl]aminosulfonyl]-1-methyl-1H-indole-2-carboxylic acid, methyl ester	25%
50	anhydrous sodium sulfate	10%
	crude calcium ligninsulfonate	5%
55	sodium alkyl naphthalenesulfonate	1%
	calcium/magnesium bentonite	59%

60 The ingredients are blended, hammer-milled and then moistened with about 12% water. The mixture is extruded as cylinders about 3 mm diameter which are cut to produce pellets about 3 mm long. These may be used directly after drying, or the dried pellets may be crushed to pass a U.S.S. No. 20 sieve (0.84 mm openings). The granules held on a U.S.S. No. 40 sieve (0.42 mm openings) may be packaged for use and the fines recycled.

65

0 070 698

Example 12

Oil Suspension

5	3-[[[4,6-dimethoxypyrimidin-2-yl]aminocarbonyl]amino-sulfonyl]-1-methyl-1H-indole-2-carboxylic acid, methyl ester	25%
	polyoxyethylene sorbitol hexaoleate	5%
10	highly aliphatic hydrocarbon oil	70%

The ingredients are ground together in a sand mill until the solid particles have been reduced to under about 5 microns. The resulting thick suspension may be applied directly, but preferably after being extended with oils or emulsified in water.

Example 13

Wettable Powder

15	N-[[[4,6-dimethoxypyrimidin-2-yl]aminocarbonyl]-3-methyl-1H-indole-2-sulfonamide	20%
20	sodium alkyl naphthalenesulfonate	4%
	sodium ligninsulfonate	4%
25	low viscosity methyl cellulose	3%
	attapulgate	69%

The ingredients are thoroughly blended. After grinding in a hammer-mill to produce particles essentially all below 100 microns, the material is rebended and sifted through a U.S.S. No. 50 sieve (0.3 mm opening) and packaged.

Example 14

Low Strength Granule

35	N-[[[4-methyl-6-methoxypyrimidin-2-yl]aminocarbonyl]-3-methyl-1H-indole-2-sulfonamide	1%
	N,N-dimethylformamide	9%
40	attapulgate granules	90%
	(U.S.S. 20—40 sieve 0.84 mm—0.42 mm)	

The active ingredient is dissolved in the solvent and the solution is sprayed upon dedusted granules in a double cone blender. After spraying of the solution has been completed, the blender is allowed to run for a short period and then the granules are packaged.

Example 15

Aqueous Suspension

50	N-[[[4,6-dimethylpyrimidin-2-yl]aminocarbonyl]-3-methyl-1H-indole-2-sulfonamide	40%
55	polyacrylic acid thickener	0.3%
	dodecylphenol polyethylene glycol ether	0.5%
	disodium phosphate	1%
60	monosodium phosphate	0.5%
	polyvinyl alcohol	1.0%
65	water	56.7%

0 070 698

The ingredients are blended and ground together in a sand mill to produce particles essentially all under 5 microns in size.

Example 16

5 *Solution*

	N-[(4-methyl-6-methoxy-1,3,5-triazin-2-yl)aminocarbonyl]- 3-methyl-1H-indole-2-sulfonamide, sodium salt	5%
10	water	95%

The salt is added directly to the water with stirring to produce the solution, which may then be packaged for use.

Example 17

15 *Low Strength Granule*

	N-[(4,6-dimethoxy-1,3,5-triazin-2-yl)aminocarbonyl]- 3-methyl-1H-indole-2-sulfonamide	0.1%
20	attapulgit granules	99.9%
	(U.S.S. 20—40 mesh 0.84—0.42 mm)	

25 The active ingredient is dissolved in a solvent and the solution is sprayed upon dedusted granules in a double-cone blender. After spraying of the solution has been completed, the material is warmed to evaporate the solvent. The material is allowed to cool and then packaged.

Example 18

30 *Granule*

	3-[[[4-methoxy-6-methylpyrimidin-2-yl)aminocarbonyl]amino- dulgonyl]-1-methyl-1H-indole-2-carboxylic acid, methyl ester	80%
35	wetting agent	1%
	crude ligninsulfonate salt (containing 5—20% of the natural sugars)	10%
40	attapulgit clay	9%

45 The ingredients are blended and milled to pass through a 100 mesh (149 microns) screen. This material is then added to a fluid bed granulator, the air flow is adjusted to gently fluidize the material, and a fine spray of water is sprayed onto the fluidized material. The fluidization and spraying are continued until granules of the desired size range are made. The spraying is stopped, but fluidization is continued, optionally with heat, until the water content is reduced to the desired level, generally less than 1%. The material is then discharged, screened to the desired size range, generally 14—100 mesh (1410—149 microns), and packaged for use.

Example 19

50 *High Strength Concentrate*

	3-[[[4,6-dimethylpyrimidin-2-yl)aminocarbonyl]amino- sulfonyl]-1-methyl-1H-indole-2-carboxylic acid, methyl ester	99%
55	silica aerogel	0.5%
	synthetic amorphous silica	0.5%

60 The ingredients are blended and ground in a hammer-mill to produce a material essentially all passing a U.S.S. No. 50 screen (0.3 mm opening). The concentrate may be formulated further if necessary.

65

0 070 698

Example 20

Wettable Powder

5	3-[[[4,6-dimethylpyrimidin-2-yl)aminocarbonyl]amino-sulfonyl]-1-methyl-1H-indole-2-carboxylic acid, methyl ester	90%
	dioctyl sodium sulfosuccinate	0.1%
10	synthetic fine silica	9.9%

The ingredients are blended and ground in a hammer-mill to produce particles essentially all below 100 microns. The material is sifted through a U.S.S. No. 50 screen (0.3 mm) and then packaged.

Example 21

Wettable Powder

15	3-[[[4-methyl-6-methoxy-1,3,5-triazin-2-yl)aminocarbonyl]-aminosulfonyl]-1-methyl-1H-indole-2-carboxylic acid, methyl ester	40%
20	sodium ligninsulfonate	20%
	montmorillonite clay	40%

25 The ingredients are thoroughly blended, coarsely hammer-milled and then air-milled to produce particles essentially all below 10 microns in size. The material is reblended and then packaged.

Example 22

Oil Suspension

30	N-[[[4,6-dimethoxy-1,3,5-triazin-2-yl-aminocarbonyl]-3-methyl-1H-indole-2-sulfonamide	35%
35	blend of polyalcohol carboxylic esters and oil soluble petroleum sulfonates	6%
	xylene	59%

40 The ingredients are combined and ground together in a sand mill to produce particles essentially all below 5 microns. The product can be used directly, extended with oils, or emulsified in water.

Utility

The compounds of the present invention are active herbicides. They have utility for broad-spectrum pre- and/or post-emergence weed control in areas where complete control of all vegetation is desired, such as around fuel storage tanks, ammunition depots, industrial storage areas, parking lots, drive-in theaters, around billboards, highway and railroad structures. Alternatively, the subject compounds are useful for the selective weed control in crops, such as wheat, cotton and soybeans.

The rates of application for the compounds of the invention are determined by a number of factors, including their use as selective or general herbicides, the crop species involved, the types of weeds to be controlled, weather and climate, formulations selected, mode of application, amount of foliage present etc. In general terms, the subject compounds should be applied at levels of around 0.03 to 10 kg/ha, the lower rates being suggested for use on lighter soils and/or those having a low organic matter content, for selective weed control or for situations where only short-term persistence is required.

50 The compounds of the invention may be used in combination with any other commercial herbicide examples of which are those of the triazine, triazole, uracil, urea, amide, diphenylether, carbamate and bipyridylium types.

The activity of these compounds was discovered in greenhouse tests. The tests are described and the data resulting from them are shown below.

60 Test A

Seeds of crabgrass (*Digitaria* spp.), barnyard grass (*Echinochloa crusgalli*), wild oats (*Avena fatua*), cassia (*Cassia tora*), morningglory (*Ipomoea* sp.), cocklebur (*Xanthium* spp), sorghum, corn, soybean, rice, wheat and nutsedge tubers (*Cyperus rotundus*) were planted in a growth medium and treated pre-emergence with the chemicals dissolved in a non-phytotoxic solvent solution of the compounds of Table A. At the same time, cotton having five leaves (including cotyledonary ones), bush beans with the third

0 070 698

trifoliolate leaf expanding, crabgrass, barnyardgrass and wild oats with two leaves, cassia with three leaves (including cotyledonary ones), morningglory and cocklebur with four leaves (including the cotyledonary ones), sorghum and corn with four leaves, soybean with two cotyledonary leaves, rice with three leaves, wheat with one leaf, and nutsedge with three to five leaves were sprayed with a non-phytotoxic solvent solution of the compounds of Table A. Treated plants and controls were maintained in a greenhouse for sixteen days, whereupon all species were compared to controls and visually rated for response to treatment. The ratings for the compounds tested by this procedure are presented in Table A. It will be seen the compounds tested possess high herbicidal activity and that certain compounds are useful for weed control in wheat and soybeans. The ratings are based on a numerical scale extending from 0 = no injury to 10 = complete kill. The accompanying descriptive symbols have the following meanings:

G= growth retardation;

C= chlorosis/netrosis;

6Y= abscised buds or flowers;

U= unusual pigmentation;

E= emergence inhibition; and

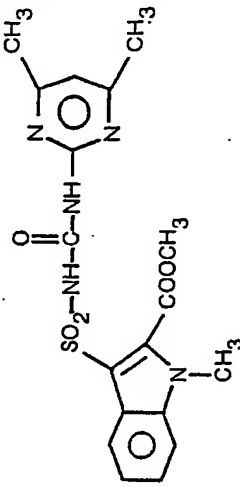
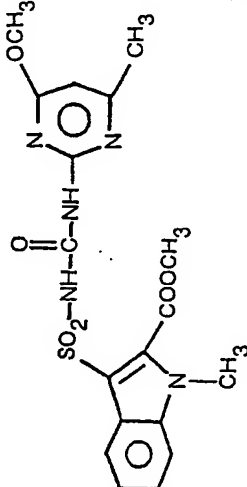
H= formative effects

U= unusual pigmentation

B= terminal bud injury.

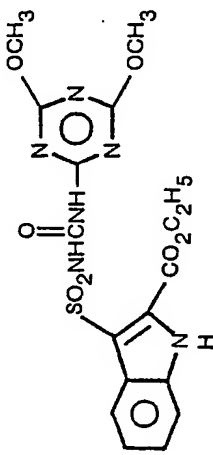
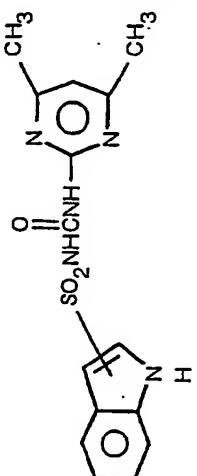
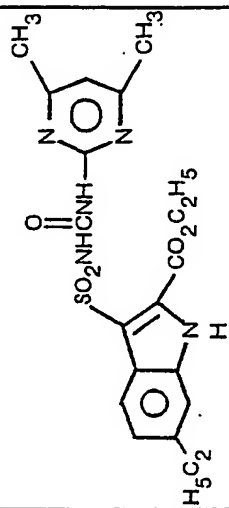
0 070 698

TABLE A

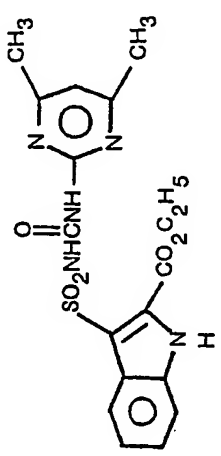
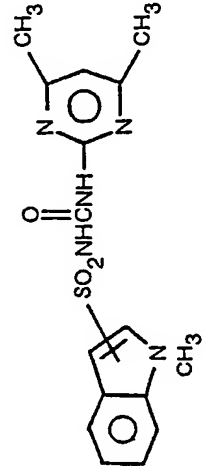
		
Rate, kg/ha	0.4	0.4
POST-EMERGENCE		
Bushbean	6C, 9G, 6Y	6C, 9G, 6Y
Cotton	6C, 9G	5C, 9G
Morningglory	2C, 4G	10C
Cocklebur	10C	10C
Cassia	6C, 9G	9C
Nutsedge	1C, 8G	4C, 8G
Crabgrass	2C, 9G	5C, 9G
Barnyardgrass	9C	10C
Wild Oats	5C, 9H	6C, 9G
Wheat	5C, 9H	5C, 9G
Corn	9C	9C
Soybean	9C	6C, 9G
Rice	2C, 9G	6C, 9G
Sorghum	9C	4C, 9G
PRE-EMERGENCE		
Morningglory	8G	9G
Cocklebur	9H	9H
Cassia	8H	8G
Nutsedge	10E	10E
Crabgrass	5C	6C, 9G
Barnyardgrass	5C	6C, 9H
Wild Oats	5C	4C, 9G
Wheat	2C	9H
Corn	10H	9G
Soybean	2C	9H
Rice	10E	10E
Sorghum	6C	5C, 9H

0 070 698

Rate, kg /ha	0.4	0.4	0.4
POST- EMERGENCE			
Bushbean	9C	7C, 9G, 6Y	9C
Cotton	9C	6C, 9H	9C
Sorghum	5C, 9G	5C, 9G	10C
Corn	9C	3C, 9G	2U, 9H
Soybean	9C	9C	6C, 9G
Wheat	5C, 9G	2C, 9G	2C, 9G
Wild Oats	5C, 9G	2C, 9G	5C, 9G
Rice	6C, 9G	6C, 9G	5C, 9G
Barnyardgrass	9C	5C, 9H	5C, 9H
Crabgrass	6C, 9G	5C, 9G	5C, 9G
Morningglory	9C	6C, 9G	3C, 9G
Cocklebur	6C, 9G	6C, 9G	9C
Cassia	6C, 9G	9C	5C, 9G
Nutsedge	4C, 9G	4C, 9G	2C, 9G
PRE - EMERGENCE			
Sorghum	7C, 9H	5C, 9G	6C, 9H
Corn	9H	9G	2C, 9G
Soybean	9H	3C, 8H	3C, 9H
Wheat	9H	1C, 9G	9H
Wild Oats	3C, 9G	4C, 9G	6C, 9G
Rice	10E	10E	5C, 9H
Barnyardgrass	6C, 9G	5C, 9H	5C, 9H
Crabgrass	4C, 9G	3C, 8G	3C, 3G
Morningglory	9G	9G	9G
Cocklebur	9H	9H	-
Cassia	9G	9G	8G
Nutsedge	9G	4C, 9G	9G

			
Rate, kg /ha	0.4	0.4	0.05
POST- EMERGENCE			
Bushbean	9C	1B	2C, 2H
Cotton	9C	1B	0
Sorghum	9C	2B	0
Corn	3C, 8H	2B	3G
Soybean	9C	1B	1H, 5G
Wheat	3U, 9G	1B	0
Wild Oats	6C, 9G	1B	0
Rice	9C	1B	1C, 5G
Barnyardgrass	5C, 9H	5B	0
Crabgrass	6C, 9G	2B	0
Morningglory	9C	3B	1C
Cocklebur	9C	2B	1C
Cassia	9C	2B	1C
Nutsedge	5C, 9G	0	0
PRE - EMERGENCE			
Sorghum	7C, 9H	2C	2C, 7G
Corn	2U, 9G	3G	2C, 6G
Soybean	8H	3G	1H
Wheat	2C, 9G	1C	3G
Wild Oats	5C, 9G	1C	8G
Rice	10E	5G	2C, 4G
Barnyardgrass	5C, 9H	6C	3G
Crabgrass	4C, 9G	0	0
Morningglory	9G	5G	2H
Cocklebur	9H	-	-
Cassia	2C, 9G	2C	1C
Nutsedge	10E	0	0

0 070 698

			
Rate, kg /ha	0.05	0.4	
POST- EMERGENCE			
Bushbean	4C, 8G, 6Y	2C, 3G	
Cotton	2C, 5G	0	
Sorghum	4C, 9G	0	
Corn	2C, 9H	0	
Soybean	3C, 8G	0	
Wheat	3C, 9H	0	
Wild Oats	2C, 9H	0	
Rice	5C, 9G	0	
Barnyardgrass	3C, 8H	0	
Crabgrass	2C, 7G	0	
Morningglory	2C, 6G	0	
Cocklebur	2C, 8G	0	
Cassia	3C	0	
Nutsedge	9G	0	
PRE - EMERGENCE			
Sorghum	2C, 9H	4G	
Corn	1C, 9H	1C, 5G	
Soybean	2C, 7H	0	
Wheat	1C, 9G	1C, 6G	
Wild Oats	3C, 9G	2C, 5G	
Rice	5C, 9H	3G	
Barnyardgrass	4C, 8H	0	
Crabgrass	1C, 6G	0	
Morningglory	1C, 4H	0	
Cocklebur	9H	0	
Cassia	4G	0	
Nutsedge	4G, 1C	0	

Test B

Two plastic bulb pans were filled with fertilized and limed Fallsington silt loam soil. One pan was planted with corn, sorghum, Kentucky bluegrass and several grassy weeds. The other pan was planted with cotton, soybeans, purple nutsedge (*Cyperus rotundus*), and several broadleaf weeds. The following grassy and broadleaf weeds were planted: crabgrass (*Digitaria sanguinalis*), barnyardgrass (*Echinochloa crusgalli*), wild oats (*Avena fatua*), johnsongrass (*Sorghum halepense*), dallisgrass (*Paspalum dilatatum*), giant foxtail (*Setaria faberii*), cheatgrass (*Bromus secalinus*), mustard (*Brassica arvensis*), cocklebur (*Xanthium pensylvanicum*), pigweed (*Amaranthus retroflexus*), morningglory (*Ipomoea hederacea*), cassia (*Cassia tora*), teaweed (*Sida spinosa*), velvetleaf (*Abutilon theophrasti*), and jimsonweed (*Datura stramonium*). A 12.5 cm diameter plastic pot was also filled with prepared soil and planted with rice and wheat. Another 12.5 cm pot was planted with sugarbeets. The above four containers were treated pre-emergence with two of the test compounds within the scope of the invention.

Twenty-eight days after treatment, the plants were evaluated and visually rated for response to the chemical treatments utilizing the rating system described previously for Test A. The data are summarized in Table B. Note that the compounds are useful for weed control in crops such as soybeans, wheat and cotton.

20

25

30

35

40

45

50

55

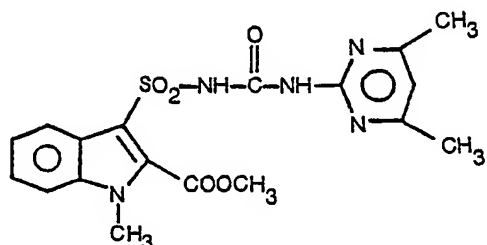
60

65

0 070 698

TABLE B

PRE-EMERGENCE ON FALLSINGTON SILT LOAM

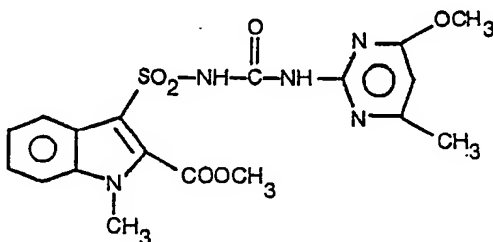


Rate, kg /ha	0.03	0.12
Crabgrass	3G	6G
Barnyardgrass	3G	7G, 3H
Sorghum	10C	10C
Wild Oats	7G, 3H	7G, 5H
Johnsongrass	4G, 2H	6G, 5H
Dallisgrass	0	4G
Giant Foxtail	3G	5G, 3H
Ky. bluegrass	7G, 5H	8G, 5H
Cheatgrass	5G, 3H	8G, 8C
Sugarbeets	7G, 3C	7G, 7C
Corn	4G, 4U	6G, 5H
Mustard	7G, 5C	9G, 9C
Cocklebur	6G, 3H	8G, 5H
Pigweed	5G	10E
Nutsedge	0	7G, 2C
Cotton	0	3G
Morningglory	0	2G
Cassia	4G	4G
Teaweed	5G	6G
Velvetleaf	2G	8G, 5H
Jimsonweed	3G	6G, 6C
Soybean	0	3G
Rice	6G, 5H	8G, 8C
Wheat	4G	5G

0 070 698

TABLE B (Continued)

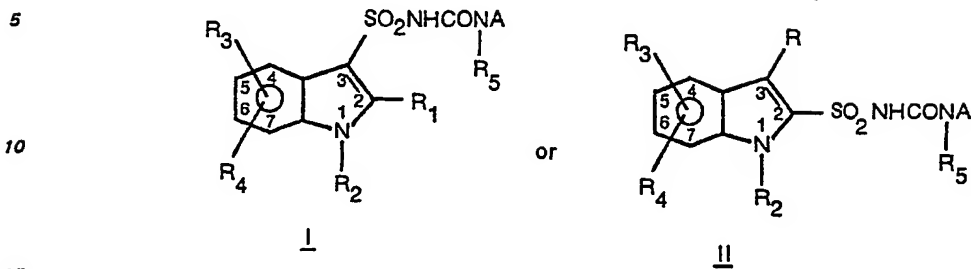
PRE-EMERGENCE ON FALLSINGTON SILT LOAM



Rate, kg /ha	0.03	0.12
Crabgrass	3G	4G
Barnyardgrass	6G	8G, 3H
Sorghum	8G, 5H	10C
Wild Oats	7G, 3H	7G, 3H
Johnsongrass	6G, 5H	7G, 5H
Dallisgrass	3G	5G
Giant Foxtail	4G, 3H	7G, 3H
Ky. bluegrass	8G, 3H	8G, 8C
Cheatgrass	4G	8G, 3H
Sugarbeets	5G, 3C	7G, 7C
Corn	5G, 5H	6G, 5H
Mustard	7G, 8C	7G, 7C
Cocklebur	4G, 3H	5G, 3H
Pigweed	5G	8G, 8C
Nutsedge	0	3G
Cotton	0	3G
Morningglory	0	4G
Cassia	0	3G
Teaweed	6G, 2C	7G, 2C
Velvetleaf	5G, 5H	7G, 5H
Jimsonweed	3G	6G, 2C
Soybean	0	4G, 2C
Rice	8G, 5H	8G, 5H
Wheat	2C	3G, 3C

Claims for the Contracting States: BE CH DE FR GB IT LI LU NL SE

1. A compound of the formula:



where

R is H, C₁—C₄ alkyl, (CH₂)_mCO₂R₉, CH₂OCH₂H₅, SO₂R₁₀, CHO, SO₂NR₁₁R₁₂, CH₂N(CH₃)₂ or CH₂OCH₃;

R₁ is H, C₁—C₄ alkyl, CO₂R₆, C(O)NR₇R₈, C(O)R₁₀, SO₂R₁₀, or SO₂NR₁₁R₁₂;

R_2 is H, C_1-C_3 alkyl or $SO_2C_6H_5$;

R₃ is H, F, Cl, Br, C₁—C₃ alkyl, C₁—C₃ alkoxy or NO₂;

R_4 is H, Cl or Br;

R_5 is H or CH_3 ;

R₆ is C₁—C₄ alkyl, C₃—C₄ alkenyl, CH₂CH₂Cl or CH₂CH₂OCH₃;

R₇ and R₈ are independently H or C₁—C₄ alkyl, provided that the total number of carbon atoms is less than or equal to 4:

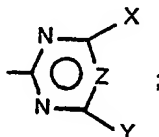
R₉ H or C₁—C₃ alkyl;

R₁₀ is C₁—C₃ alkyl;

R₁₁ and R₁₂ are independently C₁—C₃ alkyl, provided that the total number of carbon atoms is less than or equal to 4;

m is 0, 1 or 2;

A is



X is CH₃ or OCH₃;

Y is CH_3 , OCH_3 , OC_2H_5 , CH_2OCH_3 , Cl , H , C_2H_5 or $\text{N}(\text{CH}_3)_2$; and

Z is CH or N;

provided that

1) when R_2 is $\text{SO}_2\text{C}_6\text{H}_5$, then R_1 and R are H or $\text{C}_1\text{--C}_4$ alkyl.

2) when Y is Cl, then Z is CH; and

3) when m is 0, then R_9 is C_1 — C_3 alkyl.

2. A compound of Claim 1, Formula I where R_1 is H, C_1-C_3 alkyl, CO_2R_6 , $C(O)NR_7R_8$, $SO_2NR_{11}R_{12}$ or SO_2R_{10} ; and R_5 is H.

3. A compound of Claim 2 where R₃ and R₄ are H.

4. A compound of Claim 3 where R_2 is H or CH_3 .

5. A compound of Claim 4 where R_1 is H, CO_2CH_3 , SO_2CH_3 or $SO_2N(CH_3)_2$.

6. A compound of Claim 5 where Y is CH₃, OCH₃, OC₂H₅, CH₂OCH₃ or Cl.

7. A compound of Claim 1, Formula II where R is H, C₁—C₃ alkyl, (CH₂)_mCO₂R₉, SO₂R₁₀ or SO₂NR₁₁R₁₂, and R₅ is H.

8. A compound of Claim 7 where R₃ and R₄ are H.

9. A compound of Claim 8 where R_2 is H or CH_3 .

10. A compound of Claim 9 where R is H, CH₃ or (CH₂)_mCO₂—(C₁—C₃ alkyl).

11. A compound of Claim 10 where Y is CH₃, OCH₃, OC₂H₅, CH₂OCH₃ or Cl.

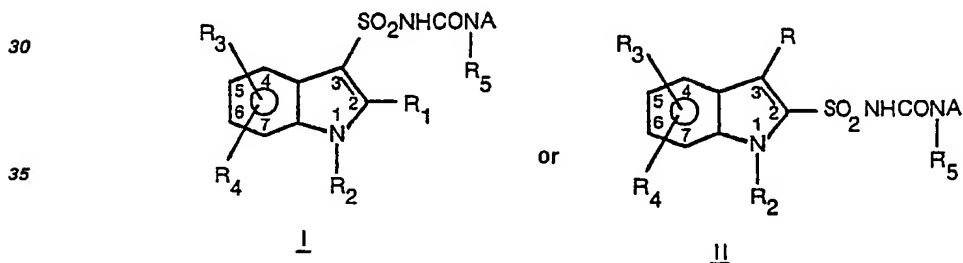
12. The compound of Claim 1, 3-[[[4,6-dimethoxypyrimidin-2-yl]aminocarbonyl]aminosulfonyl]-1-methyl-1H-indole-2-carboxylic acid, methyl ester.

13. The compound of Claim 1, 3-[[[4,6-dimethylpyrimidin-2-yl]aminocarbonyl]aminosulfonyl]-1-methyl-1H-indole-2-carboxylic acid, methyl ester.

14. The compound of Claim 1, 3-[[[4-methoxy-6-methylpyrimidin-2-yl]aminocarbonyl]aminosulfonyl]-1-methyl-1H-indole-2-carboxylic acid, methyl ester.

15. The compound of Claim 1, 3-[[[4,6-dimethoxy-1,3,5-triazin-2-yl]aminocarbonyl]aminosulfonyl]-1-methyl-1H-indole-2-carboxylic acid, methyl ester.

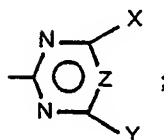
16. The compound of Claim 1, 3-[[[4,6-dimethyl-1,3,5-triazin-2-yl]aminocarbonyl]aminosulfonyl]-1-methyl-1H-indole-2-carboxylic acid, methyl ester.
17. The compound of Claim 1, 3-[[[4-methyl-6-methoxy-1,3,5-triazin-2-yl]aminocarbonyl]aminosulfonyl]-1-methyl-1H-indole-2-carboxylic acid, methyl ester.
18. The compound of Claim 1, N-[[[4,6-dimethoxypyrimidin-2-yl]aminocarbonyl]-3-methyl-1H-indole-2-sulfonamide.
19. The compound of Claim 1, N-[[[4,6-dimethylpyrimidin-2-yl]aminocarbonyl]-3-methyl-1H-indole-2-sulfonamide.
20. The compound of Claim 1, N-[[[4-methyl-6-methoxypyrimidin-2-yl]aminocarbonyl]-3-methyl-1H-indole-2-sulfonamide.
21. The compound of Claim 1, N-[[[4,6-dimethoxy-1,3,5-triazin-2-yl]aminocarbonyl]-3-methyl-1H-indole-2-sulfonamide.
22. The compound of Claim 1, N-[[[4,6-dimethyl-1,3,5-triazin-2-yl]aminocarbonyl]-3-methyl-1H-indole-2-sulfonamide.
23. The compound of Claim 1, N-[[[4-methyl-6-methoxy-1,3,5-triazin-2-yl]aminocarbonyl]-3-methyl-1H-indole-2-sulfonamide.
24. The compound of Claim 1, 3-[[[4,6-dimethoxypyrimidin-2-yl]aminocarbonyl]aminosulfonyl]-1H-indole-2-carboxylic acid, ethyl ester.
25. The compound of Claim 1, 3-[[[4,6-dimethoxy-1,3,5-triazin-2-yl]aminocarbonyl]aminosulfonyl]-1H-indole-2-carboxylic acid, ethyl ester.
26. The compound of Claim 1, 3-[[[4-methoxy-6-methyl-1,3,5-triazin-2-yl]aminocarbonyl]aminosulfonyl]-1H-indole-2-carboxylic acid, ethyl ester.
27. The compound of Claim 1, 3-[[[4-methoxy-6-methylpyrimidin-2-yl]aminocarbonyl]aminosulfonyl]-1H-indole-2-carboxylic acid, ethyl ester.
28. The compound of Claim 1, 3-[[[4,6-dimethylpyrimidin-2-yl]aminocarbonyl]aminosulfonyl]-1H-indole-2-carboxylic acid, ethyl ester.
29. A compound selected from



40 where

- R is H, C₁—C₄ alkyl or (CH₂)_mCO₂R₉;
- R₁ is H, C₁—C₄ alkyl, CO₂R₆, C(O)NR₇R₈, CN, C(O)R₉ or SO₂R₁₀;
- R₂ is H, C₁—C₃ alkyl;
- R₃ is H, F, Cl, Br, C₁—C₃ alkyl, C₁—C₃ alkoxy or NO₂;
- R₄ is H, Cl or Br;
- R₅ is H or CH₃;
- R₆ is C₁—C₄ alkyl, C₃—C₄ alkenyl, CH₂CH₂Cl or CH₂CH₂OCH₃;
- R₇ and R₈ are independently H or C₁—C₄ alkyl, provided that the total number of carbon atoms is less than or equal to 4;
- R₉ is H or C₁—C₃ alkyl;
- R₁₀ is C₁—C₃ alkyl;
- m is 1 or 2;
- A is

55



60

- X is CH₃ or OCH₃;
- Y is CH₃, OCH₃, OC₂H₅, CH₂OCH₃, Cl or H; and
- Z is CH or N;
- provided that
- when Y is Cl then Z is CH.

65

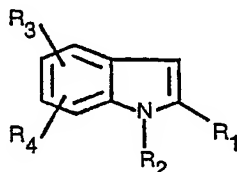
30. A composition suitable for controlling the growth of undesired vegetation comprising an effective amount of a herbicidal compound and at least one of the following: surfactant, solid or liquid diluent, characterised in that said herbicidal compound comprises a compound of any one of the preceding claims.

31. A method for controlling the growth of undesired vegetation by applying to the locus to be protected an effective amount of a herbicidal compound, characterised in that said herbicidal compound comprises a compound of any one of claims 1 to 28.

32. A process for the preparation of a compound as claimed in claim 1 which comprises:—

a) for the preparation of a compound of formula I, the reaction of a compound of the formula:

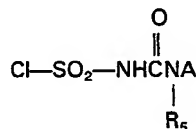
10



15

(wherein R_1 , R_2 , R_3 and R_4 are as defined in claim 1) with a compound of the formula:

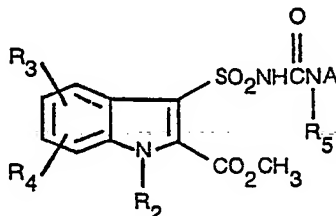
20



25 (wherein A and R_5 are as defined in claim 1); or

b) for the preparation of a compound of formula I wherein R_1 represents the group $-\text{CONR}_7\text{R}_8$ (in which R_7 and R_8 are as defined in claim 1), the reaction of a compound of the formula:

30



35

(wherein A, R_2 , R_3 , R_4 and R_5 are as defined in claim 1) with a compound of the formula:—

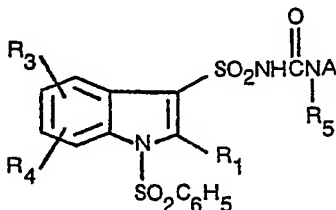
40



(wherein R_7 and R_8 are as defined in claim 1) or

45 c) for the preparation of a compound of formula I in which R_1 is H or C_1-C_4 alkyl and R_2 is H, the alkaline hydrolysis of a compound of the formula:

50

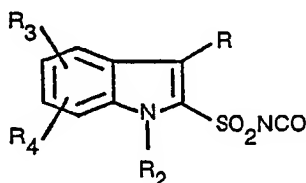


55

(wherein R_1 is H or C_1-C_4 alkyl and A, R_3 , R_4 and R_5 are as defined in claim 1); or

d) for the preparation of a compound of formula II, the reaction of a compound of the formula:

60



65

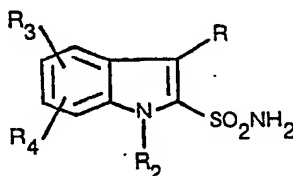
(wherein R, R₂, R₃ and R₄ are as defined in claim 1) with a compound of the formula:



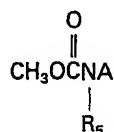
V

(wherein A and R₅ are as defined in claim 1); or

e) for the preparation of a compound of formula II, the reaction of a compound of the formula:



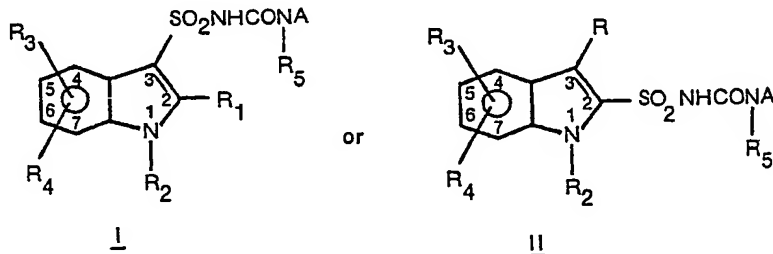
(wherein R, R₂, R₃ and R₄ are as defined in claim 1) with a compound of the formula:



(wherein A and R₅ are as defined in claim 1) in the presence of an appropriate catalyst.

Claims for the Contracting State: AT

1. A process for the preparation of a compound of formula:



where

R is H, C₁—C₄ alkyl, (CH₂)_mCO₂R₉, CH₂OC₂H₅, SO₂R₁₀, CHO, SO₂NR₁₁R₁₂, CH₂N(CH₃)₂ or CH₂OCH₃;

R₁ is H, C₁—C₄ alkyl, CO₂R₆, C(O)NR₇R₈, C(O)R₁₀, SO₂R₁₀, or SO₂NR₁₁R₁₂;

R₂ is H, C₁—C₃ alkyl or SO₂C₆H₅;

R₃ is H, F, Cl, Br, C₁—C₃ alkyl, C₁—C₃ alkoxy or NO₂;

R₄ is H, Cl or Br;

R₅ is H or CH₃;

R₆ is C₁—C₄ alkyl, C₃—C₄ alkenyl, CH₂CH₂Cl or CH₂CH₂OCH₃;

R₇ and R₈ are independently H or C₁—C₄ alkyl, provided that the total number of carbon atoms is less than or equal to 4;

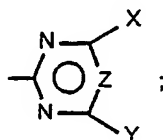
R₉ H or C₁—C₃ alkyl;

R₁₀ is C₁—C₃ alkyl;

R₁₁ and R₁₂ are independently C₁—C₃ alkyl, provided that the total number of carbon atoms is less than or equal to 4;

m is 0, 1 or 2;

A is



X is CH₃ or OCH₃;

0 070 698

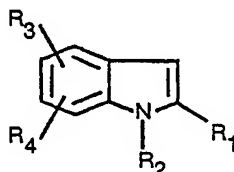
Y is CH₃, OCH₃, OC₂H₅, CH₂OCH₃, Cl, H, C₂H₅ or N(CH₃)₂; and
Z is CH or N;

provided that

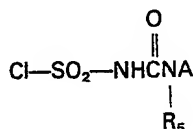
- 1) when R₂ is SO₂C₆H₅, then R₁ and R are H or C₁—C₄ alkyl.
- 2) when Y is Cl, then Z is CH; and
- 3) when m is 0, then R₉ is C₁—C₃ alkyl.

which process comprises:—

a) for the preparation of a compound of formula I, the reaction of a compound of the formula:

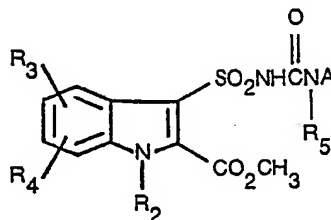


(wherein R₁, R₂, R₃ and R₄ are as defined in claim 1) with a compound of the formula:



(wherein A and R₅ are as defined in claim 1); or

b) for the preparation of a compound of formula I wherein R₁ represents the group —CONR₇R₈ (in which R₇ and R₈ are as defined in claim 1), the reaction of a compound of the formula:

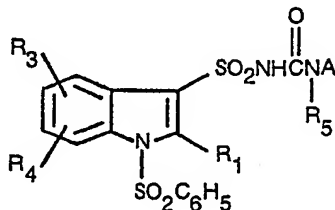


(wherein A, R₂, R₃, R₄ and R₅ are as defined in claim 1) with a compound of the formula:—



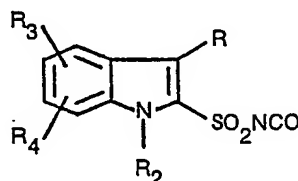
(wherein R₇ and R₈ are as defined in claim 1) or

c) for the preparation of a compound of formula I in which R₁ is H or C₁—C₄ alkyl and R₂ is H, the alkaline hydrolysis of a compound of the formula:



(wherein R₁ is H or C₁—C₄ alkyl and A, R₃, R₄ and R₅ are as defined in claim 1); or

d) for the preparation of a compound of formula II, the reaction of a compound of the formula:



VI

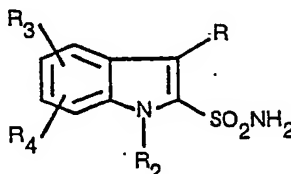
(wherein R, R₂, R₃ and R₄ are as defined in claim 1) with a compound of the formula:



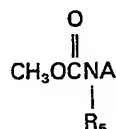
V

(wherein A and R₅ are as defined in claim 1); or

e) for the preparation of a compound of formula II, the reaction of a compound of the formula:



(wherein R, R₂, R₃ and R₄ are as defined in claim 1) with a compound of the formula:



(wherein A and R₅ are as defined in claim 1) in the presence of an appropriate catalyst.

2. A process as claimed in claim 1 for the preparation of a compound of formula I where R₁ is H, C₁—C₃ alkyl, CO₂R₆, C(O)NR₇R₈, SO₂NR₁₁R₁₂ or SO₂R₁₀; and R₅ is H.

3. A process as claimed in claim 2 where R₃ and R₄ are H.

4. A process as claimed in claim 3 wherein R₂ is H or CH₃.

5. A process as claimed in claim 4 wherein R₁ is H, CO₂CH₃, SO₂CH₃ or SO₂N(CH₃)₂.

6. A process as claimed in claim 5 where Y is CH₃, OCH₃, OC₂H₅, CH₂OCH₃ or Cl.

7. A process as claimed in claim 1 for the preparation of a compound of formula II where R is H, C₁—C₃ alkyl, (CH₂)_mCO₂R₉, SO₂R₁₀ or SO₂NR₁₁R₁₂; and R₅ is H.

8. A process as claimed in claim 7 where R₃ and R₄ are H.

9. A process as claimed in claim 8 where R₂ is H or CH₃.

10. A process as claimed in claim 9 where R is H, CH₃ or (CH₂)_mCO₂—(C₁—C₃ alkyl).

11. A process as claimed in claim 10 where Y is CH₃, OCH₃, OC₂H₅, CH₂OCH₃ or Cl.

12. The process of claim 1 wherein the product is a compound selected from:

3-[[[(4,6-dimethoxypyrimidin-2-yl)aminocarbonyl]aminosulfonyl]-1-methyl-1H-indole-2-carboxylic acid, methyl ester;

3-[[[(4,6-dimethylpyrimidin-2-yl)aminocarbonyl]aminosulfonyl]-1-methyl-1H-indole-2-carboxylic acid, methyl ester.

3-[[[(4-methoxy-6-methylpyrimidin-2-yl)aminocarbonyl]aminosulfonyl]-1-methyl-1H-indole-2-carboxylic acid, methyl ester;

3-[[[(4,6-dimethoxy-1,3,5-triazin-2-yl)aminocarbonyl]aminosulfonyl]-1-methyl-1H-indole-2-carboxylic acid, methyl ester;

3-[[[(4,6-dimethyl-1,3,5-triazin-2-yl)aminocarbonyl]aminosulfonyl]-1-methyl-1H-indole-2-carboxylic acid, methyl ester;

3-[[[(4-methyl-6-methoxy-1,3,5-triazin-2-yl)aminocarbonyl]aminosulfonyl]-1-methyl-1H-indole-2-carboxylic acid, methyl ester;

N-[(4,6-dimethoxypyrimidin-2-yl)aminocarbonyl]-3-methyl-1H-indole-2-sulfonamide;

N-[(4,6-dimethylpyrimidin-2-yl)aminocarbonyl]-3-methyl-1H-indole-2-sulfonamide;

N-[(4-methyl-6-methoxypyrimidin-2-yl)aminocarbonyl]-3-methyl-1H-indole-2-sulfonamide;

N-[(4,6-dimethyl-1,3,5-triazin-2-yl)aminocarbonyl]-3-methyl-1H-indole-2-sulfonamide;

N-[(4-methyl-6-methoxy-1,3,5-triazin-2-yl)aminocarbonyl]-3-methyl-1H-indole-2-sulfonamide;

3-[[[(4,6-dimethoxypyrimidin-2-yl)aminocarbonyl]aminosulfonyl]-1H-indole-2-carboxylic acid, ethyl ester;

3-[[[(4,6-dimethoxy-1,3,5-triazin-2-yl)aminocarbonyl]aminosulfonyl]-1H-indole-2-carboxylic acid, ethyl ester;

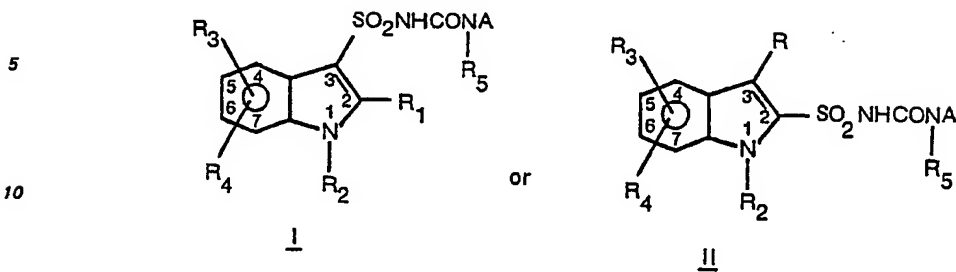
3-[[[(4-methoxy-6-methyl-1,3,5-triazin-2-yl)aminocarbonyl]aminosulfonyl]-1H-indole-2-carboxylic acid, ethyl ester;

3-[[[(4-methoxy-6-methylpyrimidin-2-yl)aminocarbonyl]aminosulfonyl]-1H-indole-2-carboxylic acid, ethyl ester; and

3-[[[(4,6-dimethylpyrimidin-2-yl)aminocarbonyl]aminosulfonyl]-1H-indole-2-carboxylic acid, ethyl ester.

0 070 698

13. The process of claim 1 wherein the product is a compound selected from



where

- 15 R is H, C₁—C₄ alkyl or (CH₂)_mCO₂R₉;
R₁ is H, C₁—C₄ alkyl, CO₂R₆, C(O)NR₇R₈, CN, C(O)R₉ or SO₂R₁₀;
R₂ is H, C₁—C₃ alkyl;
R₃ is H, F, Cl, Br, C₁—C₃ alkyl, C₁—C₃ alkoxy or NO₂;
R₄ is H, Cl or Br;
20 R₅ is H or CH₃;
R₆ is C₁—C₄ alkyl, C₃—C₄ alkenyl, CH₂CH₂Cl or CH₂CH₂OCH₃;
R₇ and R₈ are independently H or C₁—C₄ alkyl, provided that the total number of carbon atoms is less than or equal to 4;
R₉ is H or C₁—C₃ alkyl;
25 R₁₀ is C₁—C₃ alkyl;
m is 1 or 2;
A is



- 35 X is CH₃ or OCH₃;
Y is CH₃, OCH₃, OC₂H₅, CH₂OCH₃, Cl or H; and
Z is CH or N;

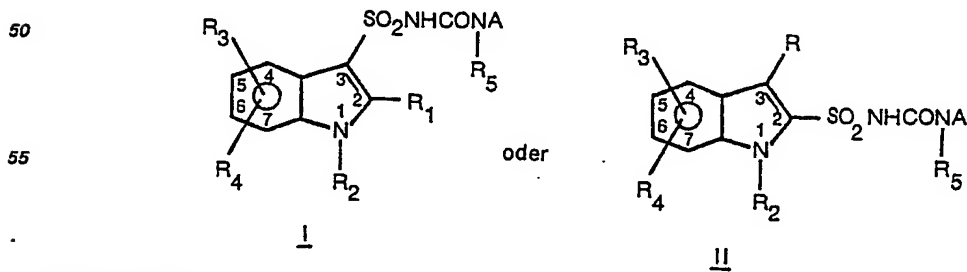
provided that

when Y is Cl then Z is CH.

14. A method for controlling the growth of undesired vegetation which comprises applying to the locus
40 to be protected an effective amount of a herbicidal compound, characterised in that said herbicidal
compound comprises a compound of formula I or II as defined in any one of claims 1 to 12.
15. A method as claimed in claim 14 wherein said compound of formula I or II is a compound as defined
in claim 13.

45 Patentansprüche für die Vertragsstaaten: BE CH DE FR GB IT LI LU NL SE

1. Eine Verbindung der Formel



60 worin bedeutet

- $R_1 H, C_1-C_4\text{-Alkyl}, (CH_2)_m CO_2 R_9, CH_2 OC_2 H_5, SO_2 R_{10}, CHO, SO_2 NR_{11} R_{12}, CH_2 N(CH_3)_2 \text{ oder } CH_2 OCH_3;$
 $R_1 H, C_1-C_4\text{-Alkyl}, CO_2 R_6, C(O)NR_7 R_8, C(O)R_{10}, SO_2 R_{10} \text{ oder } SO_2 NR_{11} R_{12};$
 $R_2 H, C_1-C_3\text{-Alkyl oder } SO_2 C_6 H_5;$
 $R_3 H, F, Cl, Br, C_1-C_3\text{-Alkyl}, C_1-C_3\text{-Alkoxy oder } NO_2;$
 $R_4 H, Cl \text{ oder } Br;$

R_5 H oder CH_3 ;
 R_6 C_1 — C_4 -Alkyl; C_3 — C_4 -Alkenyl, $\text{CH}_2\text{CH}_2\text{Cl}$ oder $\text{CH}_2\text{CH}_2\text{OCH}_3$;
 R_7 und R_8 unabhängig H oder C_1 — C_4 -Alkyl, vorausgesetzt, dass die Gesamtzahl von C-Atomen weniger als oder gleich 4 beträgt;

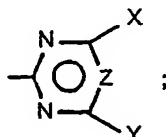
5 R_9 H oder C_1 — C_3 -Alkyl;

R_{10} C_1 — C_3 -Alkyl;

R_{11} und R_{12} unabhängig C_1 — C_3 -Alkyl, vorausgesetzt, dass die Gesamtzahl von C-Atomen weniger als oder gleich 4 beträgt;

m 0, 1 oder 2;

10 A



15

X CH_3 oder OCH_3 ;

Y CH_3 , OCH_3 , OC_2H_5 , CH_2OCH_3 , Cl, H, C_2H_5 oder $\text{N}(\text{CH}_3)_2$; und

Z CH oder N;

20 vorausgesetzt, dass

1) wenn R_2 $\text{SO}_2\text{C}_6\text{H}_5$ ist, R_1 und R H oder C_1 — C_4 -Alkyl sind;

2) wenn Y Cl ist, Z CH ist; und

3) wenn m 0 ist, R_9 C_1 — C_3 -Alkyl ist.

2. Eine Verbindung nach Anspruch 1, Formel I, worin R_1 H, C_1 — C_3 -Alkyl, CO_2R_6 , $\text{C}(\text{O})\text{NR}_7\text{R}_8$, $\text{SO}_2\text{NR}_{11}\text{R}_{12}$
 25 oder SO_2R_{10} und R_5 H ist.

3. Eine Verbindung nach Anspruch 2, worin R_3 und R_4 H sind.

4. Eine Verbindung nach Anspruch 3, worin R_2 H oder CH_3 ist.

5. Eine Verbindung nach Anspruch 4, worin R_1 H, CO_2CH_3 , SO_2CH_3 oder $\text{SO}_2\text{N}(\text{CH}_3)_2$ ist.

6. Eine Verbindung nach Anspruch 5, worin Y CH_3 , OCH_3 , OC_2H_5 , CH_2OCH_3 oder Cl ist.

7. Eine Verbindung nach Anspruch 1, Formel II, worin R H, C_1 — C_3 -Alkyl, $(\text{CH}_2)_m\text{CO}_2\text{R}_9$, SO_2R_{10} oder
 30 $\text{SO}_2\text{NR}_{11}\text{R}_{12}$ und R_5 H ist.

8. Eine Verbindung nach Anspruch 7, worin R_3 und R_4 H sind.

9. Eine Verbindung nach Anspruch 8, worin R_2 H oder CH_3 ist.

10. Eine Verbindung nach Anspruch 9, worin R H, CH_3 oder $(\text{CH}_2)_m\text{CO}_2$ —(C_1 — C_3 -Alkyl) ist.

11. Eine Verbindung nach Anspruch 10, worin Y CH_3 , OCH_3 , OC_2H_5 , CH_2OCH_3 oder Cl ist.

12. Die Verbindung nach Anspruch 1, nämlich 3-[[[4,6-Dimethoxypyrimidin-2-yl)aminocarbonyl]aminosulfonyl]-1-methyl-1H-indol-2-carbonsäure-methylester.

13. Die Verbindung nach Anspruch 1, nämlich 3-[[[4,6-Dimethylpyrimidin-2-yl)aminocarbonyl]aminosulfonyl]-1-methyl-1H-indol-2-carbonsäure-methylester.

14. Die Verbindung nach Anspruch 1, nämlich 3-[[[4-Methoxy-6-methylpyrimidin-2-yl)aminocarbonyl]aminosulfonyl]-1-methyl-1H-indol-2-carbonsäure-methylester.

15. Die Verbindung nach Anspruch 1, nämlich 3-[[[4,6-Dimethoxy-1,3,5-triazin-2-yl)aminocarbonyl]aminosulfonyl]-1-methyl-1H-indol-2-carbonsäure-methylester.

16. Die Verbindung nach Anspruch 1, nämlich 3-[[[4,6-Dimethyl-1,3,5-triazin-2-yl)aminocarbonyl]aminosulfonyl]-1-methyl-1H-indol-2-carbonsäure-methylester.

17. Die Verbindung nach Anspruch 1, nämlich 3-[[[4-Methyl-6-methoxy-1,3,5-triazin-2-yl)aminocarbonyl]aminosulfonyl]-1-methyl-1H-indol-2-carbonsäure-methylester.

18. Die Verbindung nach Anspruch 1, nämlich N-[(4,6-Dimethoxypyrimidin-2-yl)aminocarbonyl]-3-methyl-1H-indol-2-sulfonamid.

19. Die Verbindung nach Anspruch 1, nämlich N-[(4,6-Dimethylpyrimidin-2-yl)aminocarbonyl]-3-methyl-1H-indol-2-sulfonamid.

20. Die Verbindung nach Anspruch 1, nämlich N-[(4-Methyl-6-methoxypyrimidin-2-yl)aminocarbonyl]-3-methyl-1H-indol-2-sulfonamid.

21. Die Verbindung nach Anspruch 1, nämlich N-[(4,6-Dimethoxy-1,3,5-triazin-2-yl)aminocarbonyl]-3-methyl-1H-indol-2-sulfonamid.

22. Die Verbindung nach Anspruch 1, nämlich N-[(4,6-Dimethyl-1,3,5-triazin-2-yl)aminocarbonyl]-3-methyl-1H-indol-2-sulfonamid.

23. Die Verbindung nach Anspruch 1, nämlich N-[(4-Methyl-6-methoxy-1,3,5-triazin-2-yl)aminocarbonyl]-3-methyl-1H-indol-2-sulfonamid.

24. Die Verbindung nach Anspruch 1, nämlich 3-[[[4,6-Dimethoxypyrimidin-2-yl)aminocarbonyl]aminosulfonyl]-1H-indol-2-carbonsäure-ethylester.

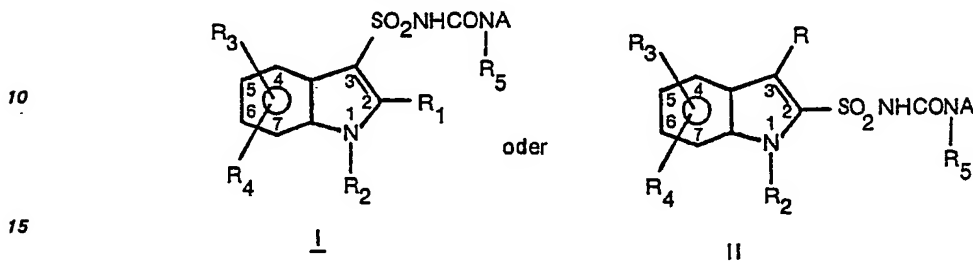
25. Die Verbindung nach Anspruch 1, nämlich 3-[[[4,6-Dimethoxy-1,3,5-triazin-2-yl)aminocarbonyl]aminosulfonyl]-1H-indol-2-carbonsäure-ethylester.

26. Die Verbindung nach Anspruch 1, nämlich 3-[[[4,6-Methoxy-6-methyl-1,3,5-triazin-2-yl)aminocarbonyl]aminosulfonyl]-1H-indol-2-carbonsäure-ethylester.

27. Die Verbindung nach Anspruch 1, nämlich 3-[[[4-Methoxy-6-methylpyrimidin-2-yl]aminocarbonyl]aminosulfonyl]-1H-indol-2-carbonsäure-ethylester.

28. Die Verbindung nach Anspruch 1, nämlich 3-[[[4,6-Dimethylpyrimidin-2-yl]aminocarbonyl]aminosulfonyl]-1H-indol-2-carbonsäure-ethylester.

5 29. Eine Verbindung, ausgewählt aus



worin bedeutet

R H, C₁—C₄-Alkyl oder (CH₂)_mCO₂R₉;

R₁ H, C₁—C₄-Alkyl, CO₂R₆, C(O)NR₇R₈, CN, C(O)R₉ oder SO₂R₁₀;

R₂ H, C₁—C₃-Alkyl;

R_3 H, F, Cl, Br, C_1 — C_3 -Alkyl, C_1 — C_3 -Alkoxy oder NO_2 ;

R_4 H, Cl oder Br;

R_5 H oder CH_3 :

R_6 C₁—C₄-Alkyl, C₃—C₄-Alkenyl, CH₂CH₂Cl oder CH₂CH₂OCH₃;

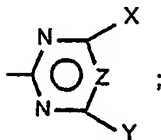
R₇ und R₈ unabhängig H oder C₁–C₄-Alkyl, vorausgesetzt, dass die Gesamtzahl von C-Atomen weniger als oder gleich 4 beträgt;

R_9 H oder C_1-C_3 -Alkyl;

R₁₀ C₁—C₃-Alkyl;

m 1 oder 2;

A



X CH₃ oder OCH₃:

Y CH₃, OCH₃, OC₂H₅, CH₂OCH₃, Cl oder H; und

Z CH oder N;

40 vorausgesetzt, dass

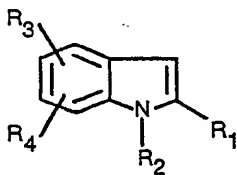
wenn Y Cl ist, Z CH ist.

30. Eine Zusammensetzung, geeignet für die Kontrolle des Wachstums unerwünschter Vegetation, enthaltend eine wirksame Menge einer herbiziden Verbindung und mindestens einen der folgenden Zusätze: oberflächenaktives Mittel, festes oder flüssiges Verdünnungsmittel, dadurch gekennzeichnet, dass die herbizide Verbindung eine Verbindung nach einem der vorhergehenden Ansprüche enthält.

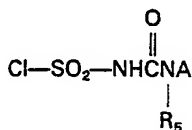
31. Verfahren zur Kontrolle des Wachstums unerwünschter Vegetation durch Aufbringung einer wirksamen Menge einer herbiziden Verbindung auf die zu schützende Stelle, dadurch gekennzeichnet, dass die herbizide Verbindung eine Verbindung nach einem der Ansprüche 1 bis 28 enthält.

32. Verfahren zur Herstellung einer Verbindung gemäss Anspruch 1, bei dem man

50 a) für die Herstellung einer Verbindung der Formel I eine Verbindung der Formel

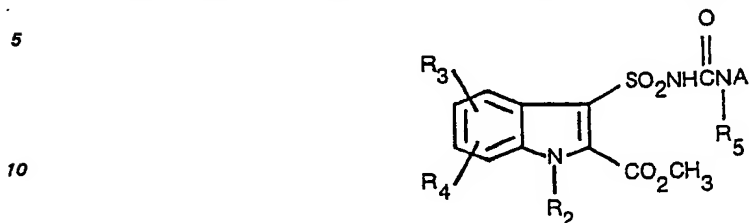


62 (worin R_1 , R_2 , R_3 und R_4 wie in Anspruch 1 definiert sind) mit einer Verbindung der Formel



0 070 698

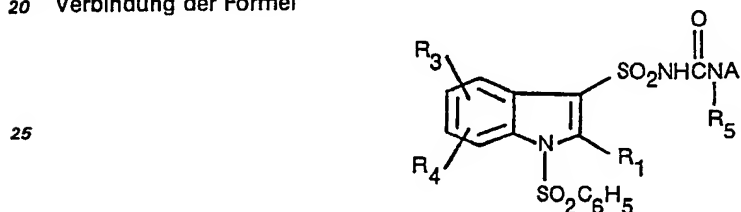
(worin A und R₅ wie in Anspruch 1 definiert sind) umgesetzt; oder
b) für die Herstellung einer Verbindung der Formel I, worin R₁ die Gruppe —CONR₇R₈ (in welcher R₇ und R₈ wie in Anspruch 1 definiert sind) bedeutet, eine Verbindung der Formel



(worin A, R₂, R₃, R₄ und R₅ wie in Anspruch 1 definiert sind) mit einer Verbindung der Formel

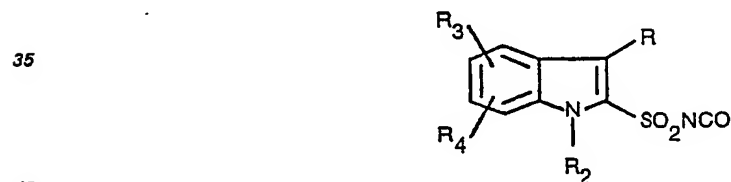


(worin R₇ und R₈ wie in Anspruch 1 definiert sind) umgesetzt; oder
c) für die Herstellung einer Verbindung der Formel I, in der R₁ H oder C₁—C₄-Alkyl und R₂ H ist, eine
20 Verbindung der Formel



30 (worin R₁ H oder C₁—C₄-Alkyl und A, R₃, R₄ und R₅ wie in Anspruch 1 definiert sind) der alkalischen Hydrolyse unterwirft; oder

d) für die Herstellung einer Verbindung der Formel II eine Verbindung der Formel



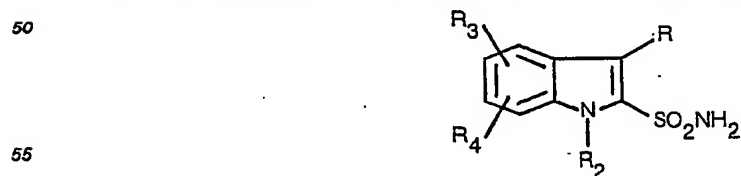
VI

(worin R, R₂, R₃ und R₄ wie in Anspruch 1 definiert sind) mit einer Verbindung der Formel



V

(worin A und R₅ wie in Anspruch 1 definiert sind) umgesetzt; oder
e) für die Herstellung einer Verbindung der Formel II eine Verbindung der Formel



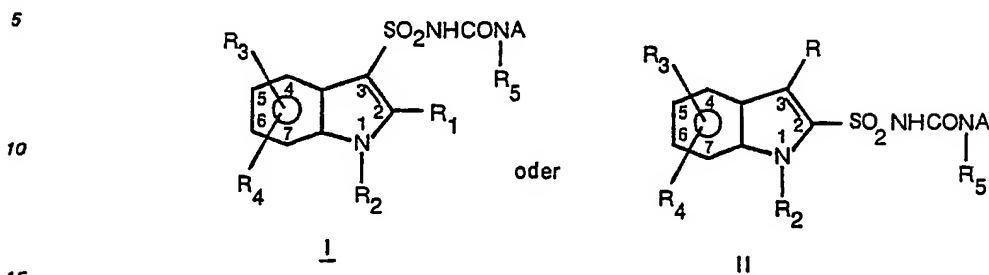
(worin R, R₂, R₃ und R₄ wie in Anspruch 1 definiert sind) mit einer Verbindung der Formel



(worin A und R₅ wie in Anspruch 1 definiert sind) in Anwesenheit eines zweckentsprechenden Katalysators
65 umgesetzt.

Patentansprüche für den Vertragsstaat: AT

1. Verfahren zur Herstellung einer Verbindung der Formel



15. worin bedeutet

R H, C₁—C₄-Alkyl, (CH₂)_mCO₂R₉, CH₂OC₂H₅, SO₂R₁₀, CHO, SO₂NR₁₁R₁₂, CH₂N(CH₃)₂ oder CH₂OCH₃;

R₁ H, C₁—C₄-Alkyl, CO₂R₆, C(O)NR₇R₈, C(O)R₁₀, SO₂R₁₀ oder SO₂NR₁₁R₁₂;

R₂ H, C₁—C₃-Alkyl oder SO₂C₆H₅;

20 R₃ H, F, Cl, Br, C₁—C₃-Alkyl, C₁—C₃-Alkoxy oder NO₂;

R₄ H, Cl oder Br;

R₅ H oder CH₃;

R₆ C₁—C₄-Alkyl; C₃—C₄-Alkenyl, CH₂CH₂Cl oder CH₂CH₂OCH₃;

25 R₇ und R₈ unabhängig H oder C₁—C₄-Alkyl, vorausgesetzt, dass die Gesamtzahl von C-Atomen weniger als oder gleich 4 beträgt;

R₉ H oder C₁—C₃-Alkyl;

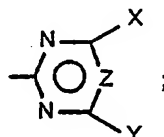
R₁₀ C₁—C₃-Alkyl;

R₁₁ und R₁₂ unabhängig C₁—C₃-Alkyl, vorausgesetzt, dass die Gesamtzahl von C-Atomen weniger als oder gleich 4 beträgt;

30 m 0, 1 oder 2;

A

35



X CH₃ oder OCH₃;

40 Y CH₃, OCH₃, OC₂H₅, CH₂OCH₃, Cl, H, C₂H₅ oder N(CH₃)₂; und

Z CH oder N;

vorausgesetzt, dass

1) wenn R₂ SO₂C₆H₅ ist, R₁ und R H oder C₁—C₄-Alkyl sind;

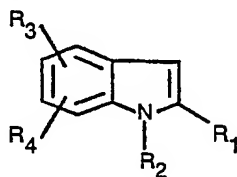
2) wenn Y Cl ist, Z CH ist; und

45 3) wenn m 0 ist, R₉ C₁—C₃-Alkyl ist,

bei dem man

a) für die Herstellung einer Verbindung der Formel I eine Verbindung der Formel

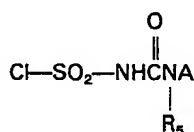
50



55

(worin R₁, R₂, R₃ und R₄ wie in Anspruch 1 definiert sind) mit einer Verbindung der Formel

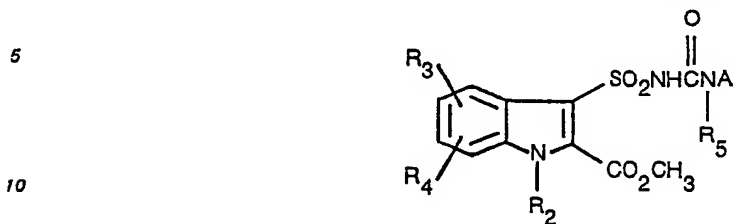
60



65 (worin A und R₅ wie in Anspruch 1 definiert sind) umgesetzt; oder

0 070 698

b) für die Herstellung einer Verbindung der Formel I, worin R_1 die Gruppe $-\text{CONR}_7\text{R}_8$ (in welcher R_7 und R_8 wie in Anspruch 1 definiert sind) bedeutet, eine Verbindung der Formel

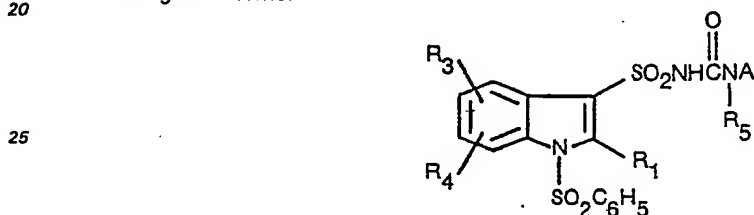


(worin R_2 , R_3 , R_4 und R_5 wie in Anspruch 1 definiert sind) mit einer Verbindung der Formel



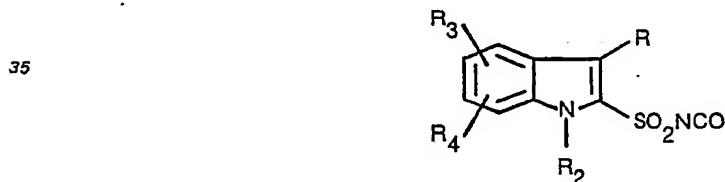
(worin R_7 und R_8 wie in Anspruch 1 definiert sind) umgesetzt; oder

c) für die Herstellung einer Verbindung der Formel I, in der R_1 H oder C_1-C_4 -Alkyl und R_2 H ist, eine Verbindung der Formel



(worin R_1 H oder C_1-C_4 -Alkyl und R_2 , R_3 , R_4 und R_5 wie in Anspruch 1 definiert sind) der alkalischen Hydrolyse unterwirft; oder

d) für die Herstellung einer Verbindung der Formel II eine Verbindung der Formel



VI

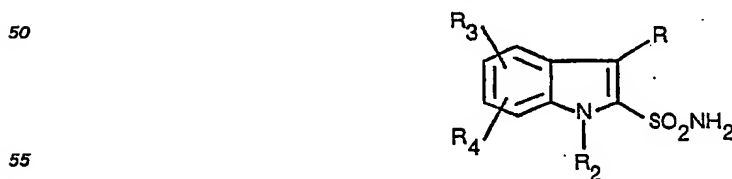
(worin R , R_2 , R_3 und R_4 wie in Anspruch 1 definiert sind) mit einer Verbindung der Formel



V

(worin A und R_5 wie in Anspruch 1 definiert sind) umgesetzt; oder

e) für die Herstellung einer Verbindung der Formel II eine Verbindung der Formel

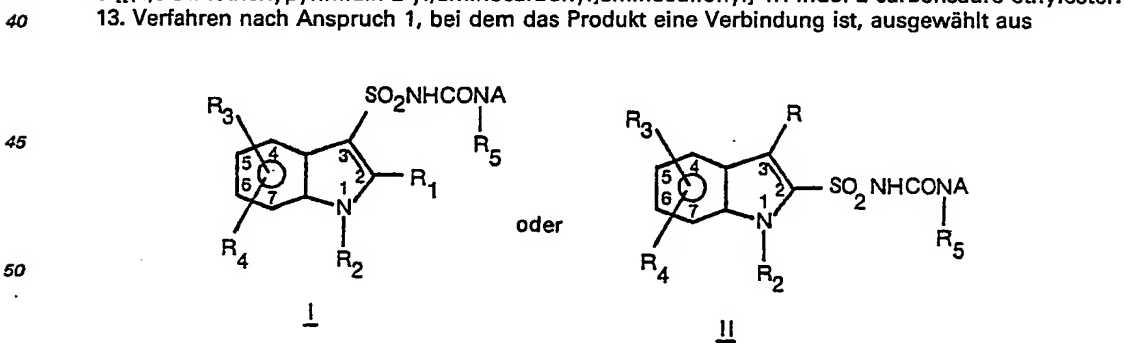


(worin R , R_2 , R_3 und R_4 wie in Anspruch 1 definiert sind) mit einer Verbindung der Formel



(worin A und R_5 wie in Anspruch 1 definiert sind) in Anwesenheit eines zweckentsprechenden Katalysators umgesetzt.

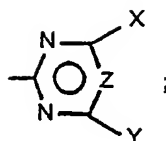
2. Verfahren nach Anspruch 1 zur Herstellung einer Verbindung der Formel I, in der R_1 H, C_1-C_3 -Alkyl, CO_2-R_6 , $C(O)NR_7R_8$, $SO_2NR_{11}R_{12}$ oder SO_2R_{10} und R_5 H ist.
 3. Verfahren nach Anspruch 2, bei dem R_3 und R_4 H sind.
 4. Verfahren nach Anspruch 3, bei dem R_2 H oder CH_3 ist.
 5. Verfahren nach Anspruch 4, bei dem R_1 H, CO_2CH_3 , SO_2CH_3 oder $SO_2N(CH_3)_2$ ist.
 6. Verfahren nach Anspruch 5, bei dem Y CH_3 , OCH_3 , OC_2H_5 , CH_2OCH_3 oder Cl ist.
 7. Verfahren nach Anspruch 1 zur Herstellung einer Verbindung der Formel II, in der R H, C_1-C_3 -Alkyl, $(CH_2)_mCO_2R_9$, SO_2R_{10} oder $SO_2NR_{11}R_{12}$ und R_5 H ist.
 8. Verfahren nach Anspruch 7, bei dem R_3 und R_4 H sind.
 9. Verfahren nach Anspruch 8, bei dem R_2 H oder CH_3 ist.
 10. Verfahren nach Anspruch 9, bei dem R H, CH_3 oder $(CH_2)_mCO_2-(C_1-C_3$ -Alkyl) ist.
 11. Verfahren nach Anspruch 10, bei dem Y CH_3 , OCH_3 , OC_2H_5 , CH_2OCH_3 oder Cl ist.
 12. Verfahren nach Anspruch 1, bei dem das Produkt eine Verbindung ist, ausgewählt aus:
 3-[[[(4,6-Dimethoxypyrimidin-2-yl)aminocarbonyl]aminosulfonyl]-1-methyl-1H-indol-2-carbonsäure-methylester;
 3-[[[(4,6-Dimethylpyrimidin-2-yl)aminocarbonyl]aminosulfonyl]-1-methyl-1H-indol-2-carbonsäure-methylester;
 - 3-[[[(4-Methoxy-6-methylpyrimidin-2-yl)aminocarbonyl]aminosulfonyl]-1-methyl-1H-indol-2-carbonsäure-methylester;
 3-[[[(4,6-Dimethoxy-1,3,5-triazin-2-yl)aminocarbonyl]aminosulfonyl]-1-methyl-1H-indol-2-carbonsäure-methylester;
 3-[[[(4,6-Dimethyl-1,3,5-triazin-2-yl)aminocarbonyl]aminosulfonyl]-1-methyl-1H-indol-2-carbonsäure-methylester;
 3-[[[(4-Methyl-6-methoxy-1,3,5-triazin-2-yl)aminocarbonyl]aminosulfonyl]-1-methyl-1H-indol-2-carbonsäure-methylester;
 N-[(4,6-Dimethoxypyrimidin-2-yl)aminocarbonyl]-3-methyl-1H-indol-2-sulfonamid;
 N-[(4,6-Dimethylpyrimidin-2-yl)aminocarbonyl]-3-methyl-1H-indol-2-sulfonamid;
 N-[(4-Methyl-6-methoxypyrimidin-2-yl)aminocarbonyl]-3-methyl-1H-indol-2-sulfonamid;
 N-[(4,6-Dimethoxy-1,3,5-triazin-2-yl)aminocarbonyl]-3-methyl-1H-indol-2-sulfonamid;
 N-[(4,6-Dimethyl-1,3,5-triazin-2-yl)aminocarbonyl]-3-methyl-1H-indol-2-sulfonamid;
 N-[(4-Methyl-6-methoxy-1,3,5-triazin-2-yl)aminocarbonyl]-3-methyl-1H-indol-2-sulfonamid;
 3-[[[(4,6-Dimethoxypyrimidin-2-yl)aminocarbonyl]aminosulfonyl]-1H-indol-2-carbonsäure-ethylester;
 3-[[[(4,6-Dimethoxy-1,3,5-triazin-2-yl)aminocarbonyl]aminosulfonyl]-1H-indol-2-carbonsäure-ethylester;
 3-[[[(4-Methoxy-6-methyl-1,3,5-triazin-2-yl)aminocarbonyl]aminosulfonyl]-1H-indol-2-carbonsäure-ethylester;
 3-[[[(4-Methoxy-6-methylpyrimidin-2-yl)aminocarbonyl]aminosulfonyl]-1H-indol-2-carbonsäure-ethylester; und
 3-[[[(4,6-Dimethoxypyrimidin-2-yl)aminocarbonyl]aminosulfonyl]-1H-indol-2-carbonsäure-ethylester.



55 worin bedeutet

- R H, C_1-C_4 -Alkyl oder $(CH_2)_mCO_2R_9$;
 R_1 H, C_1-C_4 -Alkyl, CO_2R_6 , $C(O)NR_7R_8$, CN, $C(O)R_9$ oder SO_2R_{10} ;
 R_2 H, C_1-C_3 -Alkyl;
 R_3 H, F, Cl, Br, C_1-C_3 -Alkyl, C_1-C_3 -Alkoxy oder NO_2 ;
 R_4 H, Cl oder Br;
 R_5 H oder CH_3 ;
 R_6 C_1-C_4 -Alkyl, C_3-C_4 -Alkenyl, CH_2CH_2Cl oder $CH_2CH_2OCH_3$;
 R_7 und R_8 unabhängig H oder C_1-C_4 -Alkyl, vorausgesetzt, dass die Gesamtzahl von C-Atomen weniger als oder gleich 4 beträgt;
 R_9 H oder C_1-C_3 -Alkyl;

R_{10} C_1-C_3 -Alkyl;
m 1 oder 2;
A



10 X CH_3 oder OCH_3 ;
Y CH_3 , OCH_3 , OC_2H_5 , CH_2OCH_3 , Cl oder H; und
Z CH oder N;

vorausgesetzt, dass
wenn Y Cl ist, Z CH ist.

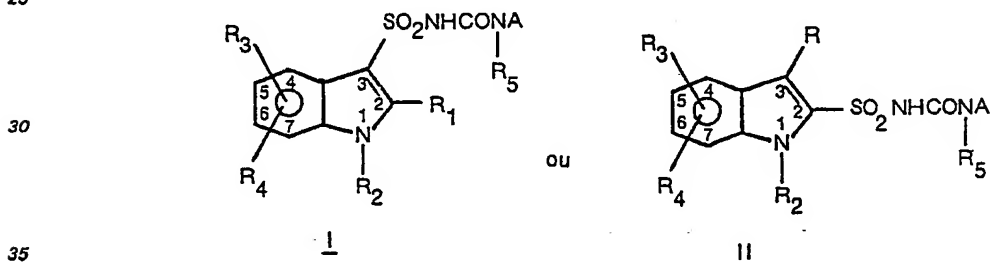
15 14. Verfahren zur Kontrolle des Wachstums unerwünschter Vegetation, bei dem man auf die zu schützende Stelle eine wirksame Menge einer herbiziden Verbindung aufbringt, dadurch gekennzeichnet, dass die herbizide Verbindung eine Verbindung der Formel I oder II enthält, wie in einem der Ansprüche 1 bis 12 definiert ist.

20 15. Verfahren nach Anspruch 14, bei dem die Verbindung der Formel I oder II eine Verbindung wie in Anspruch 13 definiert ist.

Revendications pour les Etats contractants: BE CH DE FR GB IT LI LU NL SE

1. Un composé de la formule:

25



où R est H, un radical alcoyle en C_1-C_4 , $(CH_2)_mCO_2R_9$, $CH_2OC_2H_5$, SO_2R_{10} , CHO, $SO_2NR_{11}R_{12}$, $CH_2N(CH_3)_2$ ou CH_2OCH_3 ;

40 R_1 est H, un radical alcoyle en C_1-C_4 , CO_2R_6 , $C(O)NR_7R_8$, $C(O)R_{10}$, SO_2R_{10} , ou $SO_2NR_{11}R_{12}$;

R_2 est H, un radical alcoyle en C_1-C_3 ou $SO_2C_6H_5$;

R_3 est H, F, Cl, Br, un radical alcoyle en C_1-C_3 , alcoxy en C_1-C_3 ou NO_2 .

R_4 est H, Cl ou Br;

R_5 est H ou CH_3 ;

45 R_6 est un radical alcoyle en C_1-C_4 , alcényle en C_3-C_4 , CH_2CH_2Cl ou $CH_2CH_2OCH_3$;

R_7 et R_8 sont indépendamment H ou un radical alcoyle en C_1-C_4 , avec la condition que le nombre total d'atomes de carbone doit être inférieur ou égal à 4;

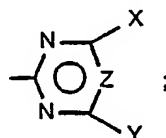
R_9 est H ou un radical alcoyle en C_1-C_3 ;

R_{10} est un radical alcoyle en C_1-C_3 ;

50 R_{11} et R_{12} sont indépendamment des radicaux alcoyle en C_1-C_3 , avec la condition que le nombre total d'atomes de carbone doit être inférieur ou égal à 4;

m est 0, 1 ou 2;

A est



60 X est CH_3 ou OCH_3 ;

Y est CH_3 , OCH_3 , OC_2H_5 , CH_2OCH_3 , Cl, H, C_2H_5 ou $N(CH_3)_2$; et

Z est CH ou N;

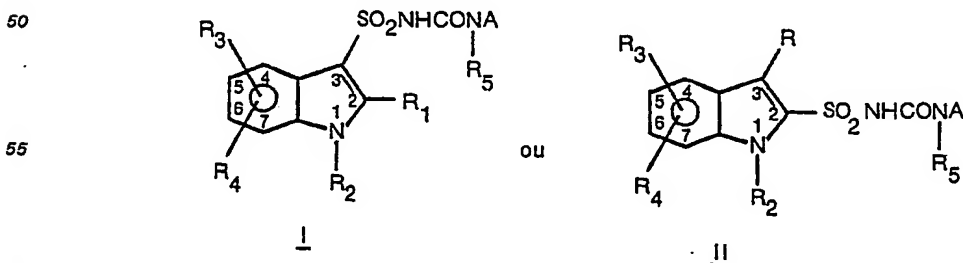
avec les conditions que:

1) quand R_2 est $SO_2C_6H_5$, alors R_1 et R sont H ou un radical alcoyle en C_1-C_4 ,

2) quand Y est Cl, alors Z est CH; et

65 3) quand m est 0, alors R_9 est un radical alcoyle en C_1-C_3 .

2. Un composé selon la revendication 1, de formule I où R est H, un radical alcoyle en C₁—C₃, CO₂R₆, C(O)NR₇R₈, SO₂NR₁₁R₁₂ ou SO₂R₁₀; et R₅ est H.
3. Un composé selon la revendication 2, dans lequel R₃ et R₄ sont H.
4. Un composé selon la revendication 3, dans lequel R₂ est H ou CH₃.
- 5 5. Un composé selon la revendication 4, dans lequel R₁ est H, CO₂CH₃, SO₂CH₃ ou SO₂N(CH₃)₂.
6. Un composé selon la revendication 5, dans lequel Y est CH₃, OCH₃, OC₂H₅, CH₂OCH₃ ou Cl.
7. Un composé selon la revendication 1, de formule II où R est H, un radical alcoyle en C₁—C₃, (CH₂)_mCO₂R₉, SO₂R₁₀ ou SO₂NR₁₁R₁₂; et R₅ est H.
8. Un composé selon la revendication 7, dans lequel R₃ et R₄ sont H.
- 10 9. Un composé selon la revendication 8, dans lequel R₂ est H ou CH₃.
10. Un composé selon la revendication 9, dans lequel R est H, CH₃ ou un radical (CH₂)_mCO₂—(alcoyle en C₁—C₃).
11. Un composé selon la revendication 10, dans lequel Y est CH₃, OCH₃, OC₂H₅, CH₂OCH₃ ou Cl.
12. Le composé selon la revendication 1, l'ester méthylique de l'acide 3-[[[4,6-diméthoxy-pyrimidin-2-yl)aminocarbonyl]aminosulfonyl]-1-méthyl-1H-indole-2-carboxylique.
13. Le composé selon la revendication 1, l'ester méthylique de l'acide 3-[[[4,6-diméthyl-pyrimidin-2-yl)aminocarbonyl]aminosulfonyl]-1-méthyl-1H-indole-2-carboxylique.
14. Le composé selon la revendication 1, l'ester méthylique de l'acide 3-[[[4-méthoxy-6-méthylpyrimidin-2-yl)aminocarbonyl]aminosulfonyl]-1-méthyl-1H-indole-2-carboxylique.
- 20 15. Le composé selon la revendication 1, l'ester méthylique de l'acide 3-[[[4,6-diméthoxy-1,3,5-triazin-2-yl)aminocarbonyl]aminosulfonyl]-1-méthyl-1H-indole-2-carboxylique.
16. Le composé selon la revendication 1, l'ester méthylique de l'acide 3-[[[4,6-diméthyl-1,3,5-triazin-2-yl)aminocarbonyl]aminosulfonyl]-1-méthyl-1H-indole-2-carboxylique.
17. Le composé selon la revendication 1, l'ester méthylique de l'acide 3-[[[4-méthyl-6-méthoxy-1,3,5-triazin-2-yl)aminocarbonyl]aminosulfonyl]-1-méthyl-1H-indole-2-carboxylique.
- 25 18. Le composé selon la revendication 1, le N-[(4,6-diméthoxy-pyrimidin-2-yl)aminocarbonyl]-3-méthyl-1H-indole-2-sulfonamide.
19. Le composé selon la revendication 1, le N-[(4,6-diméthyl-pyrimidin-2-yl)aminocarbonyl]-3-méthyl-1H-indole-2-sulfonamide.
- 30 20. Le composé selon la revendication 1, le N-[(4-méthyl-6-méthoxy-pyrimidin-2-yl)aminocarbonyl]-3-méthyl-1H-indole-2-sulfonamide.
21. Le composé selon la revendication 1, le N-[(4,6-diméthoxy-1,3,5-triazin-2-yl)aminocarbonyl]-3-méthyl-1H-indole-2-sulfonamide.
22. Le composé selon la revendication 1, le N-[(4,6-diméthyl-1,3,5-triazin-2-yl)aminocarbonyl]-3-méthyl-1H-indole-2-sulfonamide.
- 35 23. Le composé selon la revendication 1, le N-[(4-méthyl-6-méthoxy-1,3,5-triazin-2-yl)aminocarbonyl]-3-méthyl-1H-indole-2-sulfonamide.
24. Le composé selon la revendication 1, l'ester éthylique de l'acide 3-[[[4,6-diméthoxy-pyrimidin-2-yl)aminocarbonyl]aminosulfonyl]-1H-indole-2-carboxylique.
- 40 25. Le composé selon la revendication 1, l'ester éthylique de l'acide 3-[[[4,6-diméthoxy-1,3,5-triazin-2-yl)aminocarbonyl]aminosulfonyl]-1H-indole-2-carboxylique.
26. Le composé selon la revendication 1, l'ester éthylique de l'acide 3-[[[4,6-méthoxy-6-méthyl-1,3,5-triazin-2-yl)aminocarbonyl]aminosulfonyl]-1H-indole-2-carboxylique.
27. Le composé selon la revendication 1, l'ester éthylique de l'acide 3-[[[4-méthoxy-6-méthylpyrimidin-2-yl)aminocarbonyl]aminosulfonyl]-1H-indole-2-carboxylique.
- 45 28. Le composé selon la revendication 1, l'ester éthylique de l'acide 3-[[[4,6-diméthyl-pyrimidin-2-yl)aminocarbonyl]aminosulfonyl]-1H-indole-2-carboxylique.
29. Un composé choisi parmi



où

R est H, un radical alcoyle en C₁—C₄ ou (CH₂)_mCO₂R₉;
 R₁ est H, un radical alcoyle en C₁—C₄, CO₂R₆, C(O)NR₇R₈, CN, C(O)R₉ ou SO₂R₁₀;
 R₂ est H ou un radical alcoyle en C₁—C₃;
 65 R₃ est H, F, Cl, Br, un radical alcoyle en C₁—C₃, alcoxy en C₁—C₃ ou NO₂;

R_4 est H, Cl ou Br;

R_5 est H ou CH_3 ;

R_6 est un radical alcoyle en $\text{C}_1\text{—C}_4$, alcényle en $\text{C}_3\text{—C}_4$, $\text{CH}_2\text{CH}_2\text{Cl}$ ou $\text{CH}_2\text{CH}_2\text{OCH}_3$;

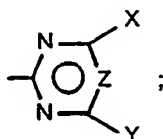
R_7 et R_8 sont indépendamment H ou un radical alcoyle en $\text{C}_1\text{—C}_4$, avec la condition que le nombre total d'atomes de carbone doit être inférieur ou égal à 4;

R_9 est H ou un radical alcoyle en $\text{C}_1\text{—C}_3$;

R_{10} est un radical alcoyle en $\text{C}_1\text{—C}_3$;

m est 1 ou 2;

A est



X est CH_3 ou OCH_3 ;

Y est CH_3 , OCH_3 , OC_2H_5 , CH_2OCH_3 , Cl ou H; et

Z est CH ou N;

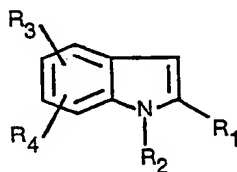
avec la condition que quand Y est Cl, alors Z est CH.

30. Une composition utilisable pour lutter contre la croissance de végétation indésirable comprenant une quantité efficace d'un composé herbicide et au moins un des ingrédients suivants: agent tensio-actif, diluant solide ou liquide, caractérisée en ce que le composé herbicide comprend un composé selon l'une quelconque des revendications précédentes.

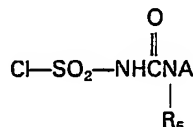
31. Un procédé de lutte contre la croissance de végétation indésirable en appliquant au lieu à protéger une quantité efficace d'un composé herbicide, caractérisé en ce que le composé herbicide comprend un composé selon l'une quelconque des revendications 1 à 28.

32. Un procédé de préparation d'un composé tel que défini dans la revendication 1, qui comprend:

a) pour la préparation d'un composé de formule I, la réaction d'un composé de la formule:

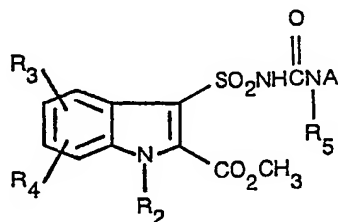


(où R_1 , R_2 , R_3 et R_4 sont tels que définis dans la revendication 1) avec un composé de la formule:



(où A et R_5 sont tels que définis dans la revendication 1); ou

b) pour la préparation d'un composé de formule I où R_1 représente le groupe $\text{—CONR}_7\text{R}_8$ (où R_7 et R_8 sont tels que définis dans la revendication 1), la réaction d'un composé de la formule:

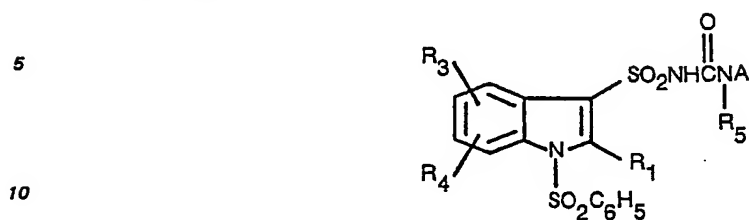


(où A, R_2 , R_3 , R_4 et R_5 sont tels que définis dans la revendication 1) avec un composé de la formule:



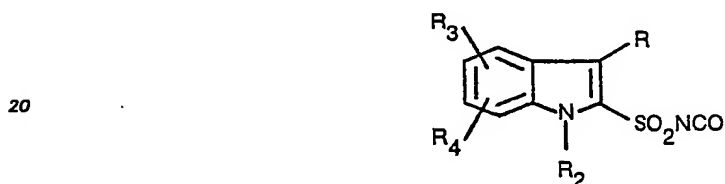
(où R_7 et R_8 sont tels que définis dans la revendication 1); ou

c) pour la préparation d'un composé de formule I où R_1 est H ou un radical alcoyle en C_1-C_4 et R_2 est H, l'hydrolyse alcaline d'un composé de la formule:



(où R₁ est H ou un radical alcoyle en C₁—C₄ et A, R₃, R₄ et R₅ sont tels que définis dans la revendication 1);
ou

d) pour la préparation d'un composé de formule II, la réaction d'un composé de la formule:



vi

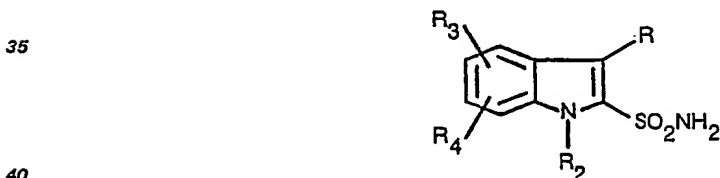
25 (où R , R_2 , R_3 et R_4 sont tels que définis dans la revendication 1) avec un composé de la formule:



V

(où A et R_5 sont tels que définis dans la revendication 1); ou

e) pour la préparation d'un composé de formule II, la réaction d'un composé de la formule:



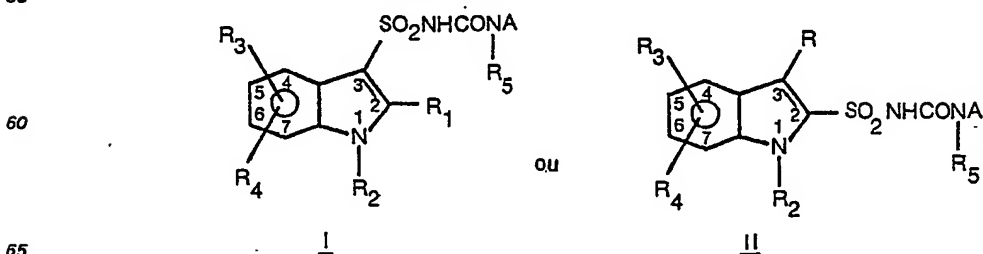
(où R , R_2 , R_3 et R_4 sont tels que définis dans la revendication 1) avec un composé de la formule



50 (où A et R₅ sont tels que définis dans la revendication 1) en présence d'un catalyseur approprié.

Revendications pour l'Etat contractant: AT

1. Un procédé pour la préparation d'un composé de formule:



où

R est H, un radical alcoyle en C_1-C_4 , $(CH_2)_mCO_2R_9$, $CH_2OC_2H_5$, SO_2R_{10} , CHO, $SO_2NR_{11}R_{12}$, $CH_2N(CH_3)_2$

ou CH_2OCH_3 ;

R_1 est H, un radical alcoyle en C_1-C_4 , CO_2R_6 , $C(O)NR_7R_8$, $C(O)R_{10}$, SO_2R_{10} , ou $SO_2NR_{11}R_{12}$;

5 R_2 est H, un radical alcoyle en C_1-C_3 ou $SO_2C_6H_5$;

R_3 est H, F, Cl, Br, un radical alcoyle en C_1-C_3 , alcoxy en C_1-C_3 ou NO_2 .

R_4 est H, Cl ou Br;

R_5 est H ou CH_3 ;

R_6 est un radical alcoyle en C_1-C_4 , alcényle en C_3-C_4 , CH_2CH_2Cl ou $CH_2CH_2OCH_3$;

10 R_7 et R_8 sont indépendamment H ou un radical alcoyle en C_1-C_4 , avec la condition que le nombre total

d'atomes de carbone doit être inférieur ou égal à 4;

R_9 est H ou un radical alcoyle en C_1-C_3 ;

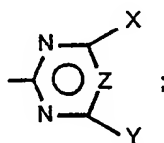
R_{10} est un radical alcoyle en C_1-C_3 ;

15 R_{11} et R_{12} sont indépendamment des radicaux alcoyle en C_1-C_3 , avec la condition que le nombre total

d'atomes de carbone doit être inférieur ou égal à 4;

m est 0, 1 ou 2;

A est



20

X est CH_3 ou OCH_3 ;

25 Y est CH_3 , OCH_3 , OC_2H_5 , CH_2OCH_3 , Cl, H, C_2H_5 ou $N(CH_3)_2$; et

Z est CH ou N;

avec les conditions que:

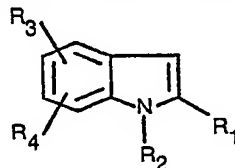
1) quand R_2 est $SO_2C_6H_5$, alors R_1 et R sont H ou un radical alcoyle en C_1-C_4 .

2) quand Y est Cl, alors Z est CH; et

30 3) quand m est 0, alors R_9 est un radical alcoyle en C_1-C_3 .

procédé qui comprend:

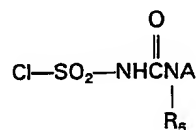
a) pour la préparation d'un composé de formule I, la réaction d'un composé de la formule:



35

40

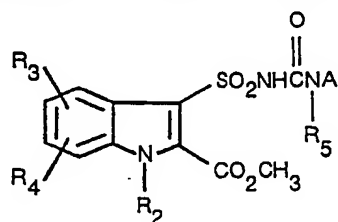
(où R_1 , R_2 , R_3 et R_4 sont tels que définis dans la revendication 1) avec un composé de la formule:



45

(où A et R_5 sont tels que définis dans la revendication 1); ou

50 b) pour la préparation d'un composé de formule I où R_1 représente le groupe $-CONR_7R_8$ (où R_7 et R_8 sont tels que définis dans la revendication 1), la réaction d'un composé de la formule:



55

60

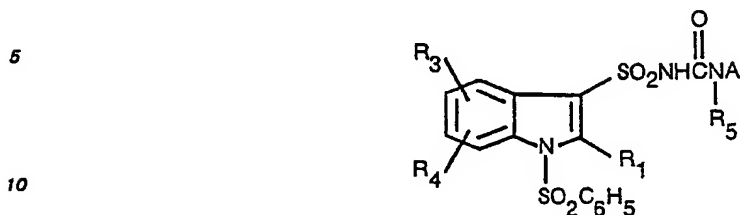
(où A, R_2 , R_3 , R_4 et R_5 sont tels que définis dans la revendication 1) avec un composé de la formule:



65 (où R_7 et R_8 sont tels que définis dans la revendication 1); ou

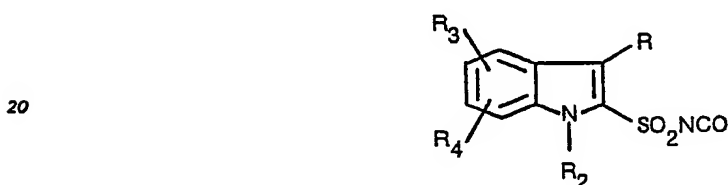
0 070 698

c) pour la préparation d'un composé de formule I où R_1 est H ou un radical alcoyle en C_1-C_4 et R_2 est H, l'hydrolyse alcaline d'un composé de la formule:



(où R_1 est H ou un radical alcoyle en C_1-C_4 et A, R_3 , R_4 et R_5 sont tels que définis dans la revendication 1); ou

15 d) pour la préparation d'un composé de formule II, la réaction d'un composé de la formule:



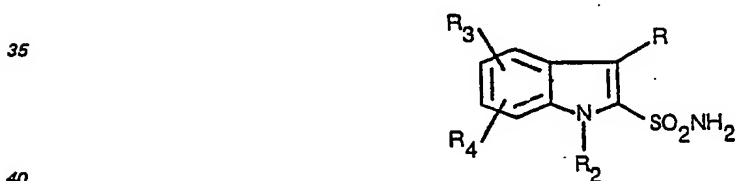
VI

25 (où R, R_2 , R_3 et R_4 sont tels que définis dans la revendication 1) avec un composé de la formule:



(où A et R_5 sont tels que définis dans la revendication 1); ou

e) pour la préparation d'un composé de formule II, la réaction d'un composé de la formule:



(où R, R_2 , R_3 et R_4 sont tels que définis dans la revendication 1) avec un composé de la formule



50 (où A et R_5 sont tels que définis dans la revendication 1) en présence d'un catalyseur approprié.

2. Un procédé selon la revendication 1 pour la préparation d'un composé de formule I où R_1 est H, un radical alcoyle en C_1-C_3 , CO_2R_6 , $C(O)NR_7R_8$, $SO_2NR_{11}R_{12}$ ou SO_2R_{10} ; et R_5 est H.

3. Un procédé selon la revendication 2, où R_3 et R_4 sont H.

4. Un procédé selon la revendication 3, où R_2 est H ou CH_3 .

55 5. Un procédé selon la revendication 4, où R_1 est H, CO_2CH_3 , SO_2CH_3 ou $SO_2N(CH_3)_2$.

6. Un procédé selon la revendication 5, où Y est CH_3 , OCH_3 , OC_2H_5 , CH_2OCH_3 ou Cl.

7. Un procédé selon la revendication 1, pour la préparation d'un composé de formule II où R est H, un radical alcoyle en C_1-C_3 , $(CH_2)_mCO_2R_9$, SO_2R_{10} ou $SO_2NR_{11}R_{12}$; et R_5 est H.

8. Un procédé selon la revendication 7, où R_3 et R_4 sont H.

60 9. Un procédé selon la revendication 8, où R_2 est H ou CH_3 .

10. Un procédé selon la revendication 9, où R est H, CH_3 ou un radical $(CH_2)_mCO_2$ -(alcoyle en C_1-C_3).

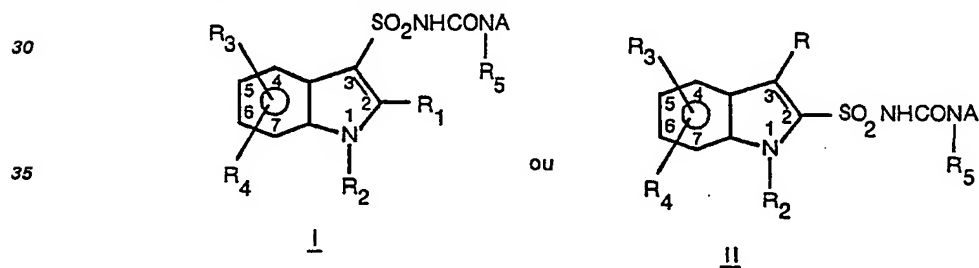
11. Un procédé selon la revendication 10, où Y est CH_3 , OCH_3 , OC_2H_5 , CH_2OCH_3 ou Cl.

12. Le procédé selon la revendication 1, dans lequel le produit est un composé choisi parmi: l'ester méthylique de l'acide 3-[[4,6-diméthoxy-pyrimidin-2-yl]aminocarbonyl] aminosulfonyl]-1-

65 méthyl-1H-indole-2-carboxylique;

- l'ester méthylique de l'acide 3-[[[(4,6-diméthylpyrimidin-2-yl)aminocarbonyl] aminosulfonyl]-1-méthyl-1H-indole-2-carboxylique;
 l'ester méthylique de l'acide 3-[[[(4-méthoxy-6-méthylpyrimidin-2-yl)-aminocarbonyl]aminosulfonyl]-1-méthyl-1H-indole-2-carboxylique;
 5 l'ester méthylique de l'acide 3-[[[(4,6-diméthoxy-1,3,5-triazin-2-yl)-aminocarbonyl]aminosulfonyl]-1-méthyl-1H-indole-2-carboxylique;
 l'ester méthylique de l'acide 3-[[[(4,6-diméthyl-1,3,5-triazin-2-yl)-aminocarbonyl]aminosulfonyl]-1-méthyl-1H-indole-2-carboxylique;
 l'ester méthylique de l'acide 3-[[[(4-méthyl-6-méthoxy-1,3,5-triazin-2-yl)-aminocarbonyl]-aminosulfonyl]-1-méthyl-1H-indole-2-carboxylique;
 10 le N-[(4,6-diméthoxy-1,3,5-triazin-2-yl)aminocarbonyl]-3-méthyl-1H-indole-2-sulfonamide;
 le N-[(4-méthyl-6-méthoxy-1,3,5-triazin-2-yl)-aminocarbonyl]-3-méthyl-1H-indole-2-sulfonamide;
 le N-[(4-méthyl-6-méthoxy-1,3,5-triazin-2-yl)-aminocarbonyl]-3-méthyl-1H-indole-2-sulfonamide;
 le N-[(4-méthyl-6-méthoxy-1,3,5-triazin-2-yl)-aminocarbonyl]-3-méthyl-1H-indole-2-sulfonamide;
 15 le N-[(4-méthyl-6-méthoxy-1,3,5-triazin-2-yl)aminocarbonyl]-3-méthyl-1H-indole-2-sulfonamide;
 le N-[(4-méthyl-6-méthoxy-1,3,5-triazin-2-yl)aminocarbonyl]-3-méthyl-1H-indole-2-sulfonamide;
 l'ester éthylique de l'acide 3-[[[(4,6-diméthoxy-1,3,5-triazin-2-yl)aminocarbonyl] aminosulfonyl]-1H-indole-2-carboxylique;
 l'ester éthylique de l'acide 3-[[[(4,6-diméthoxy-1,3,5-triazin-2-yl)aminocarbonyl] aminosulfonyl]-1H-indole-2-carboxylique;
 20 l'ester éthylique de l'acide 3-[[[(4-méthoxy-6-méthyl-1,3,5-triazin-2-yl)aminocarbonyl]-aminosulfonyl]-1H-indole-2-carboxylique;
 l'ester éthylique de l'acide 3-[[[(4-méthoxy-6-méthylpyrimidin-2-yl)aminocarbonyl] aminosulfonyl]-1H-indole-2-carboxylique; et
 25 l'ester éthylique de l'acide 3-[[[(4,6-diméthylpyrimidin-2-yl)aminocarbonyl] aminosulfonyl]-1H-indole-2-carboxylique.

13. Le procédé selon la revendication 1, dans lequel le produit est un composé choisi parmi:



- où
 R est H, un radical alcoyle en C₁—C₄ ou (CH₂)_mCO₂R₉;
 R₁ est H, un radical alcoyle en C₁—C₄, CO₂R₆, C(O)NR₇R₈, CN, C(O)R₉ ou SO₂R₁₀;
 R₂ est H ou un radical alcoyle en C₁—C₃;
 R₃ est H, F, Cl, Br, un radical alcoyle en C₁—C₃, alcoxy en C₁—C₃ ou NO₂;
 45 R₄ est H, Cl ou Br;
 R₅ est H ou CH₃;
 R₆ est un radical alcoyle en C₁—C₄, alcényle en C₃—C₄, CH₂CH₂Cl ou CH₂CH₂OCH₃;
 R₇ et R₈ sont indépendamment H ou un radical alcoyle en C₁—C₄, avec la condition que le nombre total
 d'atomes de carbone doit être inférieur ou égal à 4;
 50 R₉ est H ou un radical alcoyle en C₁—C₃;
 R₁₀ est un radical alcoyle en C₁—C₃;
 m est 1 ou 2;
 A est



- X est CH₃ ou OCH₃;
 Y est CH₃, OCH₃, OC₂H₅, CH₂OCH₃, Cl ou H; et
 Z est CH ou N;
 65 avec la condition que quand Y est Cl, alors Z est CH.

0 070 698

14. Un procédé de lutte contre la croissance de végétation indésirable en appliquant au lieu à protéger une quantité efficace d'un composé herbicide, caractérisé en ce que le composé herbicide comprend un composé de formule I ou II tel que défini dans l'une quelconque des revendications 1 à 12.

5 15. Un procédé selon la revendication 14, dans lequel le composé de formule I ou II est un composé tel que défini dans la revendication 13.

10

15

20

25

30

35

40

45

50

55

60

65